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AFRPL-TR-82-12

TWR-30863

DEVELOPMENT OF COMPOSITE CASE SALVAGE PROCEDURES
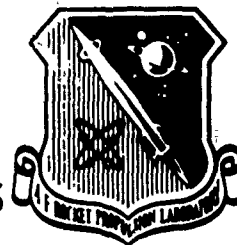
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APRIL 1982

FINAL REPORT FOR PERIOD 15 OCTOBER 1980 - 15 APRIL 1982

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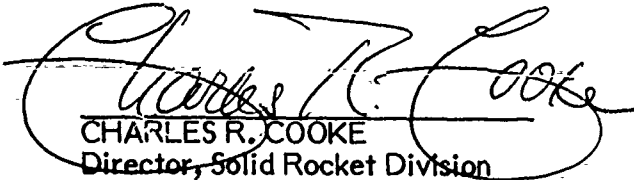
NOTICES

FOREWORD

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REPORT DOCUMENTATION PAGE	READ INSTRUCTIONS BEFORE COMPLETING FORM

1. REPORT NUMBER	2. GOVT ACCESSION NO.	3. RECIPIENT'S CATALOG NUMBER
AFRPL-TR-82-12	AD-B065369L	

4. TITLE (and Subtitle)	5. TYPE OF REPORT & PERIOD COVERED
Development of Composite Case Salvage Procedures	Final Technical Report 15 October - 15 April 1982
	6. PERFORMING ORG. REPORT NUMBER TWR-30863

7. AUTHOR(s)	8. CONTRACT OR GRANT NUMBER(s)
E. D. Brown	F04611-81-C-0001

9. PERFORMING ORGANIZATION NAME AND ADDRESS	10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS
Thiokol/Wasatch Division, P.O. Box 524 Brigham City, Utah 84302	305909 PH

11. CONTROLLING OFFICE NAME AND ADDRESS	12. REPORT DATE
Air Force Rocket Propulsion Laboratory/MKBB. Edwards AFB, CA 93523	April 1982
	13. NUMBER OF PAGES 318

14. MONITORING AGENCY NAME & ADDRESS(If different from Controlling Office)	15. SECURITY CLASS. (of this report)
AFPRO, Thiokol Corporation Wasatch Division P.O. Box 524 Brigham City, Utah 84302	Unclassified
	15a. DECLASSIFICATION/DOWNGRADING SCHEDULE

16. DISTRIBUTION STATEMENT (of this Report)

Distribution limited to U.S. Government Agencies only. Test and Evaluation, March 1982. Other requests for this document must be referred to AFRPL (STINFO)/TSPR, Stop 24, Edwards AFB, CA 93523.

17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, If different from Report)

Approved for public release; distribution unlimited.

18. SUPPLEMENTARY NOTES

19. KEY WORDS (Continue on reverse side if necessary and Identify by block number)

Composite Case, Salvage, Cost Model, Propellant Removal, Hydromining, Machining, Propellant Degradation

20. ABSTRACT (Continue on reverse side if necessary and Identify by block number)

A program was conducted in four phases to develop safe, cost effective process methods for salvaging and recycling composite cases from solid propellant rocket motors. Motors were evaluated, ranging in size from 4,000 to 200,000 lb, loaded with Class 1.1 or 1.3 propellant.

During Phase I, a literature search was conducted to determine the current state-of-the-art status of operations applicable to case salvage operations.

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Emphasis was placed upon propellant removal methods, insulation and case repair methods, and disposal of explosive wastes. An initial assessment was made of methods that appeared to be promising.

Computerized cost models were developed during Phase II for estimation of case salvage costs and the cost of fabricating new cases. Parameters considered in the computation of case salvage costs included the level of salvage, motor size, type of propellant, method of waste disposal, inspection and qualification requirements, the number of units to be salvaged, the production rate, and the methods utilized for propellant, liner and insulation removal.

During Phase III, laboratory studies were conducted to investigate new and existing methods for propellant and insulation removal and to evaluate the effect this processing would have on insulation and case integrity. Utilization and the effects of solvents were emphasized. Hydromining and machining were judged to be the better methods for propellant removal.

A program plan was developed during Phase IV for full-scale testing of salvage methods utilizing Government-furnished Minuteman III Stage III Motors. The program is also adaptable to salvage of other cases such as MX Stage I, II, or III motors.

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1.0 INTRODUCTION

1.1 PROGRAM OBJECTIVE

The objective of this program was to develop safe, cost effective process technologies for salvaging and recycling composite cases from solid propellant rocket motors ranging in size from 4,000 to 200,000 lb and containing Class 1.3 or 1.1 propellant.

1.2 SCOPE

This program consisted of four phases conducted during a 15 month technical effort followed by a 3 month final report period. The four phases were: (1) Technology Assessment, (2) Feasibility and Cost Studies, (3) Laboratory Studies, and (4) A Demonstration Program Plan.

During Phase I, a literature search was conducted to determine the current state-of-the-art of processing methods applicable to case salvage. Primary emphasis was upon methods of propellant and insulation removal, propellant waste disposal, and refurbishment of the case after propellant/liner/ insulation removal. Literature on associated technologies which could apply to the case salvage operation was also accumulated.

The objective of Phase II was to evaluate the feasibility of reclaiming composite cases using the technology identified in Phase I and determining the cost effectiveness versus the manufacture of new cases. Additional sources of input included (1) the existing Thiokol propulsion cost data and estimating model, (2) cost history of new case manufacture based on Thiokol's experience in composite case manufacture, and (3) cost data and processing experience from Thiokol's Minuteman III Stage III Case Salvage Test Program.

The Phase III laboratory studies consisted of propellant removal testing, insulation removal and reinstallation testing, and case structural testing. Special emphasis was placed upon testing solvents to degrade or desensitize the propellant for propellant removal and to determine the effect of the solvent on the insulation and case materials.

The Phase IV program plan development consisted of outlining a follow-on program to demonstrate the salvaging techniques selected from Phases I, II, and III using three Government-furnished Minuteman III Stage III motors.

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The program plan was generalized to include utilization of MX Stage I,
II, or III motors should they become available.

1.3 BACKGROUND

The practice of salvaging metal cases from defective or decommissioned
solid propellant rocket motors for reloading has proven to be cost effective
without degrading case reliability. In one of the earlier programs, Thiokol
reclaimed Minuteman I Stage I case/closure assemblies for reuse in Minuteman
II and III Stage I motors at a substantial cost savings for the Minuteman
weapon system. A high pressure water washout facility was built for removal
of the propellant, liner, and insulation by the hydromining method. This
process has been modified and adapted for reclamation of metal cases from
several different sized solid rocket motors. In addition, several composite
cases have been successfully salvaged during development programs by applying
the hydromining method. Results of these experiences, summarized in Table I,
showed that composite cases could be reclaimed undamaged when the insulation
was left intact in the motor.

The increased cost of filament wound structures, coupled with the long
lead time for new cases, makes case salvage an attractive alternative. Case
salvage has the potential for reducing cost and schedule lead time during
development programs, including MX and space programs, by allowing rapid,
low-cost salvage and reloading of reject motors. Case salvage also has po-
tential savings of reduced cost for retrofit of motors from existing weapons
systems.

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TABLE I

SUMMARY OF THIOKOL'S MOTOR RECLAMATION PROGRAMS SHOWING PROPELLANT REMOVAL METHODS USED AND THE NUMBER OF CASES SALVAGED

A. Thiokol Propellant Hydromining Experience

Motor	Propellant (wt)	No. Salvaged
Minuteman Stg I	44,000	320
Bomarc	3,000	22
Castor IV	21,000	16
Subroc	2,000	320
Genie	320	2,321
Other	to 8,000	14

Composite Cases

Poseidon	38,000	1
Trident-I	41,500	1
Trident-II	18,450	1
Minuteman Stg III	7,000	2

B. Thiokol Propellant Machining Experience**

Motor	Propellant Removed (wt)	No. Machined
120 In. Motor	10,300	1
156 In. Motor	23,400	1
Minuteman Stg I	400	2,973
Trident-I	210	203*
Genie	9	6,679
Star Space Motore	50	300

*Class 1.1 propellant
**Total propellant machined 1,337,000 lb

2.0 DISCUSSION

2.1 PHASE I – LITERATURE SEARCH AND TECHNOLOGY ASSESSMENT

The specific areas of search for applicable technology included:

1. A review of propellant chemistry and propellant or ingredient degradation or desensitization using solvents and/or reagents
2. Physical methods of propellant removal
3. Waste propellant disposal methods
4. Insulation removal and replacement technology
5. Case structural considerations

A summary of the results follows. A more detailed discussion of the results is presented in Part II, Appendix A with listings and abstracts of applicable articles.

2.1.1 Propellant Grain Degradation Using Solvents and/or Reagents

A literature search was conducted using the following:

Identifiers and Combinations of Key Words

Polymer		Urethane		Hydrolysis
Rubber	plus	Polyester	plus	Degradation
Elastomer		Polyether		Cleavage
				Recycling
				Reclamation
				Solvolysis

Also:

Salvage		Rubber
Reclamation		Elastomers
Degradation	plus	Solid Propellants
Machining		Nitroglycerine
Disposal		Explosive Materials

A total of 1,118 initial leads was obtained. From the review of the abstracts, 252 references were judged to be potentially applicable. Of these, only 18 were directly applicable to propellant degradation.

Utilization of solvents to degrade the propellant is applicable (1) to soften or degrade the propellant and facilitate the removal from the case and

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(2) to degrade the propellant for recovery of the ingredients as an alternate to incineration for waste disposal.

Although degradation of propellant grains to facilitate removal has not been studied in detail (most of the work has been directed toward ingredient recovery), there was ample information in the literature to substantiate further efforts. The major items to be defined during Phase III were determined to be (1) the rate of degradation of propellant grains, (2) the permeability of the propellant to solvents/reagents, and (3) the compatibility of propellant constituents with the solvent/reagents under the conditions used during removal processes.

2.1.2 Physical Methods of Propellant Removal

The literature search did not reveal any new or unusual methods of propellant removal. The two most feasible methods for propellant removal apparently are hydromining and machining.

During the literature search of propellant removal and waste disposal, 1,187 publications were listed in computer-based searches. Of these, 217 publications were judged to be apparently relevant and were reviewed. Only 52 of the above articles were judged to be directly applicable. The most pertinent abstracts, along with a discussion of historical application of hydromining and machining, are presented in Part II, Appendix A.

2.1.3 Waste Disposal

The abundant publications on waste propellant and explosive disposal indicate the concern for development of suitable methods of disposal. At the present time, the state-of-the-art method is open-pit incineration. Limited data on pilot plants for closed incineration processes, utilizing rotary kilns, fluidized beds, and wet air-oxidation are available. Other methods for disposal such as utilization of propellants as explosives and reclamation of ingredients are in experimental stages only. Cost evaluations for any method other than open-air incineration are limited to estimations.

Summaries of discussions with knowledgeable people in the industry are presented in Part II, Appendix A with abstracts and lists of articles and publications that were judged to be pertinent.

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2.1.4 Insulation Removal and Replacement Techniques

A considerable amount of literature is pertinent to insulation removal and replacement. These articles are primarily concerned with repair operations; however, the techniques used and results obtained are directly applicable to case salvage operations.

Articles reviewed support the Thiokol position that removal of the flaps and liner and replacement of the flaps are low risk, low cost procedures. These operations have been verified in new case manufacturing operation. Removal of structural insulation, particularly in the dome areas and around polar bosses, was not recommended.

A discussion of the methods of rework pertaining to insulation removal and replacement is presented in Part II, Appendix A. Lists of pertinent references and abstracts are included.

2.1.5 Case Structural Considerations

Most of the literature concerning case structural integrity was concerned with testing of new cases and the effects of aging during storage. The two references given in Part II, Appendix A, page A-73, were judged to be the most useful. A discussion of case design and fabrication technology is included in Part II, Appendix A. Results of Thiokol studies on effects of solvents or water on the resin, fiber damage, composite contamination, and multiproof testing are discussed.

Twenty-two references are listed in Part II, Appendix A which are pertinent to the effect of fluid on composite case properties. Results reported are varied: some report the original strength of the case is regained if the moisture or solvent is removed; others report little or no recovery.

One of the unanswered questions of case reclamation concerns the effect of the salvage processes on long-term aging of the salvaged case. The results of the LRSLA Program indicated that, although degradation of the burst strength of Minuteman III cases due to moisture may be reversible when dried out, other effects of aging may be sufficiently detrimental to make questionable the salvage of cases from stored motors.

Twelve references, listed in Part II, Appendix A, describe effects of fracture, fatigue, and general accumulated damage of composite cases.

In composites, a wider range of initial flaws is found than in metals.
The proof test itself is known to have potential for damage of the composite
case although tests have indicated little loss of strength in low cycle
applications where the applied stress level was kept below 80% of the static
strength.

In summary, a tremendous amount of literature is available which applies
to the question of composite case degradation. The following literature
searches were reviewed:

1. NASA Literature Search Number 32359, "Environmental Effects on Filament Wound Structures," 17 May 1976 – 89 articles

2. TRW Literature Search, "Aging of Glass Reinforced Plastics," Part of LRSLA Program – 31 extended abstracts

3. Phase I, Technology Assessment, CDRL Item 4, Contract No. F-4611-79-C-0038, submitted to AFRPL by Brunswick Corporation, 29 August 1980 – 238 abstracts and summaries

4. Thiokol Technical Library Literature Search, "Loading Mechanics, Damage Effects and Moisture and Temperature Effects on Composite Pressure Results and Rocket Motor Cases," December 1980 – 64 microfische, 102 reports

5. Computer Literature Search at LMSC, National Technical Information Service, "Effects of Environmental Conditions on Reinforced Plastics," March 1979 – 200 reports

6. Stage III Minuteman Fiberglass Aging Study, LRSLA Program – 32 papers

2.2 PHASE II – FEASIBILITY AND COST STUDIES

2.2.1 Objective

The basic purpose of Phase II was to develop a means of determining
whether composite case salvage is an attractive, cost-effective alternative
to new case fabrication.

The basic goals were to:

1. Establish parametric cost equations
2. Establish cost screening parameters
3. Establish cost equation coefficients
4. Computerize a model that predicts the case salvage costs and the new case costs for comparison

Items to be built into the model included:

1. Learning curve adjustments
2. Production sensitivities pertaining to:
    a. Quantities
    b. Rates of production
    c. Scheduling
3. Tooling, facility, and testing modifiers
4. Propellant hazard sensitivities
5. Motor size sensitivity
6. Methods of processing selected for propellant and insulation removal, waste disposal, and level of testing

2.2.2 New Case Costs

New case costs were developed by regressing costs for fabrication of new cases with various motor parameters. The best regression curve fit was obtained with the loaded grain weight (propellant, insulation, and case weight) versus the labor and material costs. The results are shown in Figure 1. The resultant equation is:

Case Cost $= 17,483 + 3.675$ (loaded chamber weight)

Deviations from this model can be expected due to factors which increase the complexity of the case such as thrust termination ports, multiple nozzles, extreme changes in length to diameter ratios, and extremely high operating pressures. As can be seen, it appears to be an extremely good correlation for the motors for which data were available.

2.2.3 Salvaged Case Costs

A cost prediction model was developed for case salvage in which the costs of various operations are calculated and the results are summed to
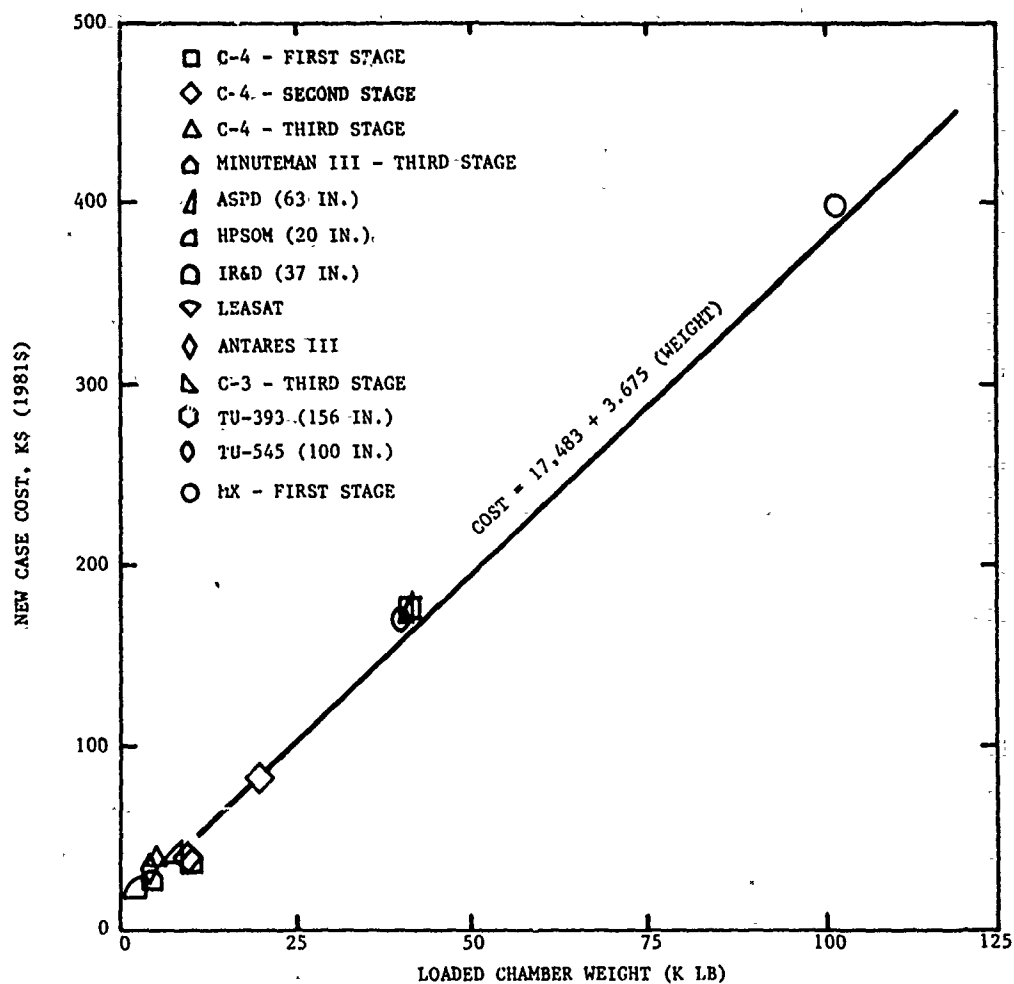
Figure 1. New case cost (labor and materials plus support) versus loaded chamber weight

determine the total cost of the salvage operation selected. The equations for estimating the costs of the various operations were based upon historical data for performance of the same or a similar operation.

The possible combinations built into the program were too numerous to possibly run all examples. The four levels of salvage operations were removal of (1) propellant, (2) propellant and liner, (3) propellant, liner, and insulation from the cylindrical section, and (4) propellant, liner, and all insulation. Methods for propellant removal were: (1) high pressure hydromining, (2) low pressure hydromining, (3) wet machining, (4) dry machining, (5) chemical degradation with hydromining, (6) chemical degradation with machining, and (7) burnout. Insulation removal methods selected were: (1) low pressure hydromining, (2) chemical degradation with hydromining, (3) chemical degradation with machining, (4) mechanical buffing or grinding, (5) manual buffing or grinding, and (6) heat and peel. Waste disposal techniques were classified into four categories: (1) open pit incineration, (2) closed process incineration, (3) ingredient reclamation, and (4) marketing propellant as explosives, fire starters, etc.

Since facilities and tooling costs could vary considerably from company to company depending upon existing facilities, the options available were: (1) to calculate costs excluding tooling and facilities, (2) to input tooling and facility costs, or (3) to allow the program to compute tooling and facility costs. The computed tooling and facility cost equations were developed based upon known costs of existing Thiokol facilities with power exponents to adjust for motor size and a multiplier to allow for propellant class.

The validity of the predictive value of the computer model was demonstrated by comparing calculated costs versus actual costs tabulated during the Thiokol Third Stage Minuteman III Program, as shown in Table II. Costs tabulated for the Minuteman III case were based on actual manhours accrued. The methods of removal for the MM III tests were: propellant by low pressure hydromining, liner by mechanical abrasion, and insulation by low pressure hydromining. Waste disposal and in-process inspection were not accounted for but were treated as overhead items. In the computed examples, no liner removal cost is calculated for a level four salvage operation, as it is assumed that the liner will be removed with the residual propellant or with

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TABLE II

SUMMARY OF COMPARISON OF MINUTEMAN III RESULTS WITH CASE SALVAGE COST ESTIMATIONS

(Minuteman III Glass Case: Propellant Weight = 7,298 lb;
Area = 38.7 ft^2; Class 1.3 Propellant; Single Motor Salvage Operation)

Task	Minuteman III[1]	Estimated Salvage Cost[2]	
Salvage Level	4	4	2
Method of Removal			
A. Propellant, Bulk	12,680	9,917	9,917
B. Propellant, Residual	---	2,606	2,606
C. Liner	2,923	---	2,478
D. Insulation, Cylindrical	3,553	2,779	---
E. Insulation, Dome	---	1,040	---
Receiving and Handling	420	645	323
Inspection – In-process	0	4,807	3,965
Inspection – Final	2,895[3]	6,761	6,761
Qualification Program	0	0	0
Reinstallation of Insulation	4,591	5,143	5,143
Waste Disposal	0	722	722
Cost Per Unit (1981 dollars) at 1 Per Month		33,800	31,900
New Case Cost (1981 dollars) at 1 Per Month		45,800	45,800
Difference (new – salvage), (1981 dollars) at 1 Per Month		12,000	13,900

Notes

1. Costs based on actual manhours charged
2. Includes estimated labor, materials, and support
3. Includes X-ray only; does not include hydrotest

the insulation. A level two salvage indicated only propellant and liner were removed; hence, there is no insulation removal cost. Reinsulation costs probably should be reduced but not eliminated since there would be minor repair and reinstallation of the flaps.

An example of the tabulation of cost estimation for various salvage operations is shown in Figure 2. The numbers of the different removal methods refer to methods listed in the preceding paragraphs and in Part II, Appendix B. This figure demonstrates that the more important drivers for the salvage cost of a particular motor are: (1) the level of salvage to be performed and (2) the method selected for propellant removal. Note that these calculations are concerned only with the anticipated costs and do not reflect potential risk of damage to the case. Hence, as shown in Figure 2, propellant removal method 7, burnout, may be more "cost effective" than the other methods, but the risk of damage to the case is judged to be too high to recommend this method. Calculations were made for motors of different sizes, and the results, presented in Figure 3, represent the approximate range of salvage costs for cases with Class 1.3 propellant as a function of the motor size.

Similar calculations were made for the same motor with Class 1.1 propellant. The results of these calculations, shown in Figure 4, are compared with the results for Class 1.3 propellant. The method for propellant removal was hydromining. The new case cost is also plotted for comparison. The results indicate that, generally, case salvage should be a profitable, cost-effective operation provided that the reclaimed case is not damaged and is suitable for reloading and final disposition.

2.2.4 Assessment of Risk

Several methods of assessing risk were examined to evaluate the feasibility of a particular salvage operation and to compare one operation to another to determine which may be more desirable. The method selected consisted of assigning risk values for each removal technique. Factors which were assigned risk values included hazard to personnel and facilities, potential damage to the insulation and case, and the feasibility factors defined as reloadability, effectiveness, and the confidence factor. Reloadability is defined as the confidence of having a reloadable case with no
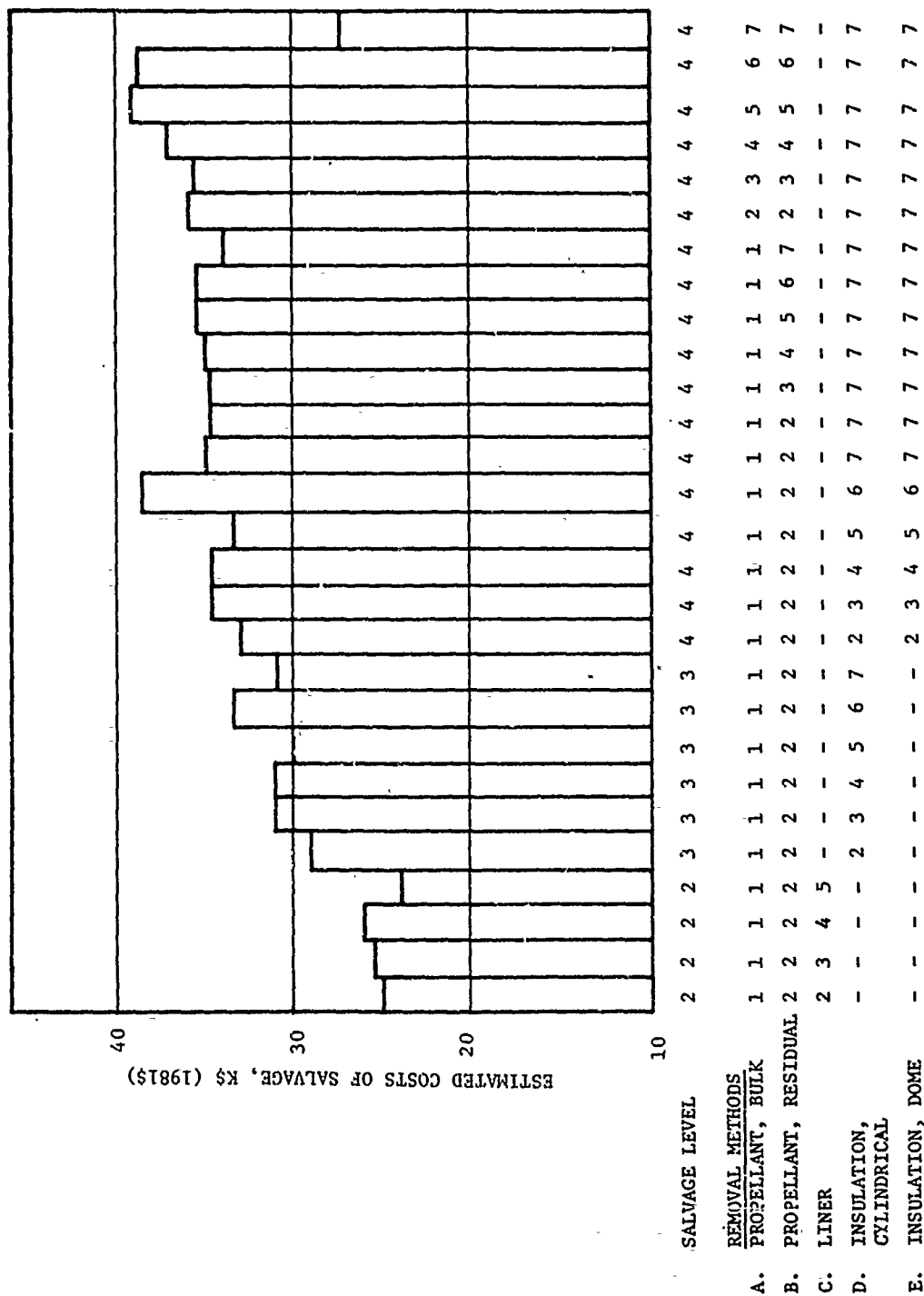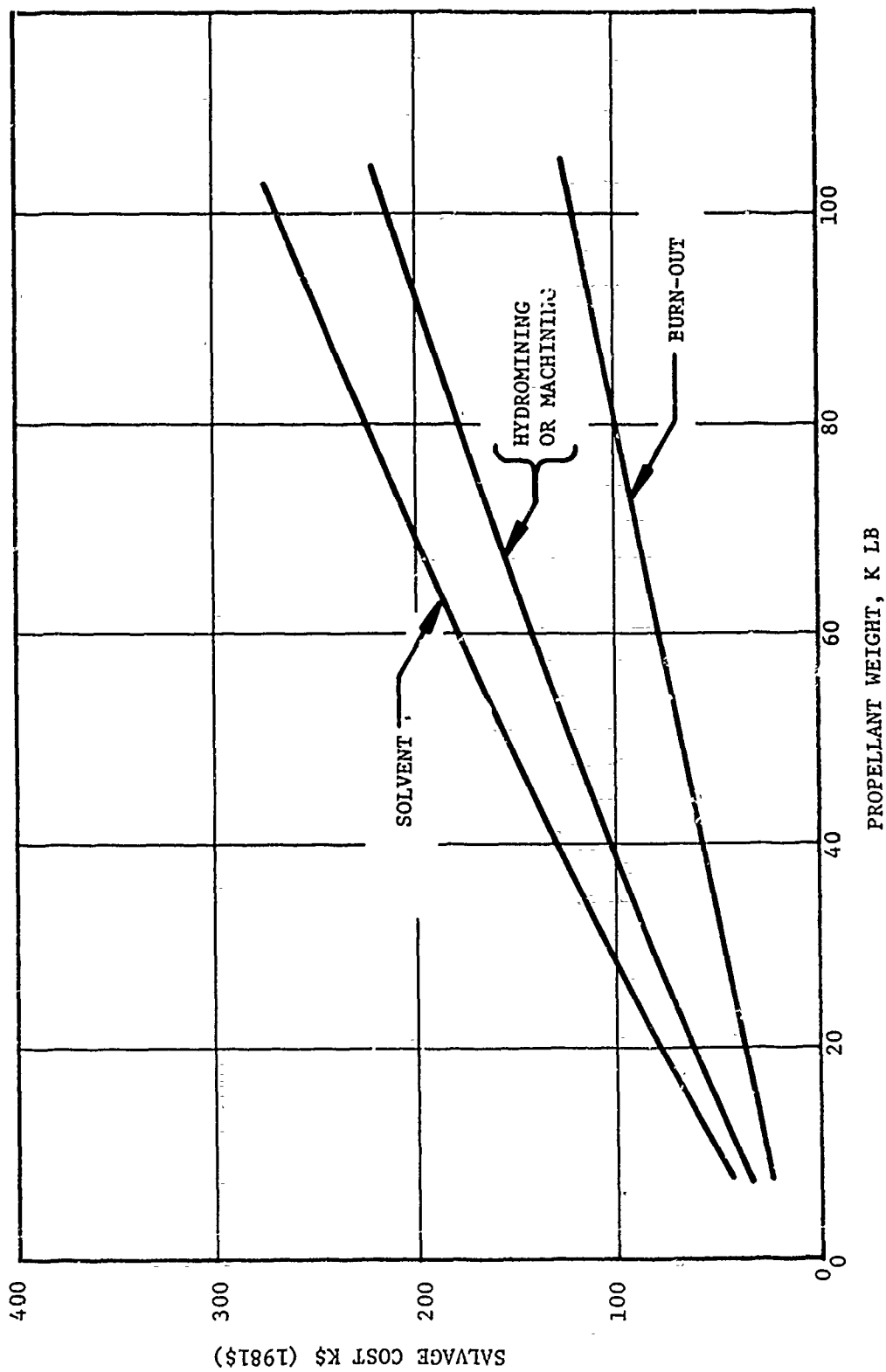
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Figure 2. Summary of estimated costs of salvage for various methods of removal and salvage levels for a Minuteman III – size case (single unit cost)

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Figure 3. Approximate range of salvage costs for cases with
Class 1.3 propellant as a function of motor size (propellant weight)

Figure 4. Comparison of new case cost with salvage costs for chambers with Class 1.3 and Class 1.1 propellants

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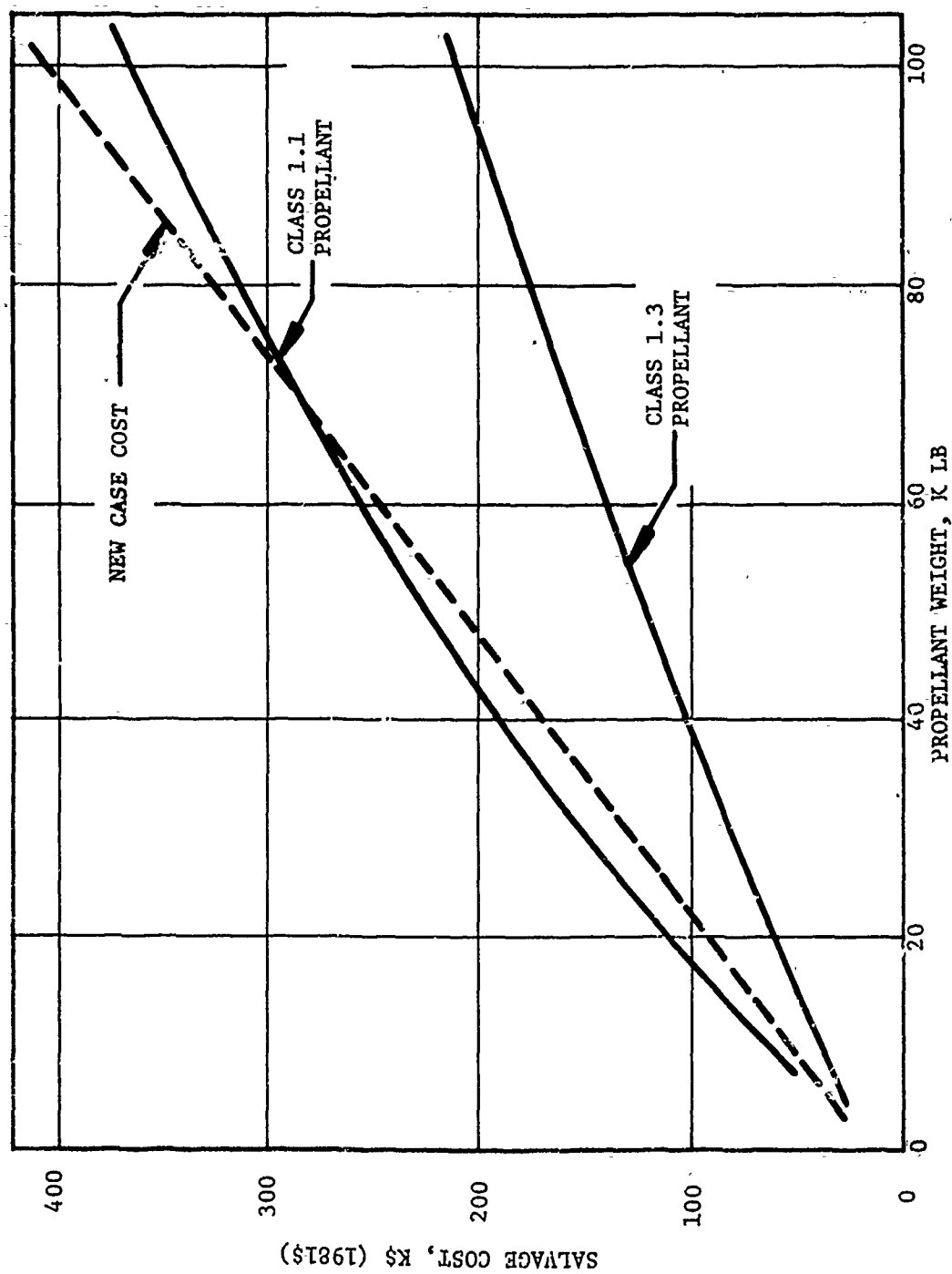
case-to-insulation debonding due to the operation. Effectiveness is the capability of the method to accomplish the operation completely in a single step operation. The confidence factor is indicative of the state of development of the technique. For a proven process or method, the risk would be low; hence, the risk value would be zero or near zero. Assignment of a value of 10 indicated the method was not acceptable. An example of the tabulated risk values for propellant removal is shown in Table III. In comparing two salvage operations, the operation which produced the lower sum of risk would be judged to be more desirable.

TABLE III

DEVELOPMENT OF RISK EVALUATION

Class 1.3 Propellant Methods of Removal	Risk Values						Feasibility	
	Safety		Damage					Confidence
	Personnel	Fac/Equip	Insulation	Case	Reloadability	Effectiveness		Factor
Hydromine, HP*	0	2	6	2	3	0		0
Hydromine, LP**	0	0	2	2	2	7		0
Machine, Wet	2	1	7	0	1	8		0
Machine, Dry	7	8	5	0	0	8		2
Chemical Degradation With Hydromining	8	4	8	8	8	9		6
Chemical Degradation With Machining	8	4	8	8	8	9		6
Burnout	4	5	10	10	10	1		8

Each method of removal assigned value between 0 – 10
0 – Indicates no problem – highly acceptable
10 – Indicates not acceptable – no possibility of this method applying

*HP = High pressure water
**LP = Low pressure, high temperature water

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2.3 PHASE III - LABORATORY STUDIES

2.3.1 Objective

The laboratory effort was designed to study and evaluate potentially cost effective propellant removal methods applicable to salvage operations. Special emphasis was placed on determining the potential risks involved in the processes, specifically (1) the hazards risk concerned with handling Class 1.1 propellants during hydromining and machining and (2) the risk of damage to motor components, case, and insulation, due to using solvents to facilitate propellant, liner, or insulation-removal operations.

2.3.2 Propellant Removal Methods

Hydromining. A total of 128 tests was conducted on six different types of propellants. Three nozzles having throat diameters of 0.055, 0.085, and 0.125 in. were used. The smallest nozzle had an elongated converging section which produced a very fine, pencil-lead-thick spray for a distance of several feet. The samples were uniformly placed 2 in. from the nozzle exit. Additional descriptions of the test and results are given in Part II, Appendix C.

The normal procedure was to increase the pressure incrementally at a given water temperature until the water cut through the 4-in. thick sample of propellant. Each test consisted of a sweep period as the water jet was rotated to cut across the propellant surface and a dwell period while the water jet impinged at one point on the propellant. During the high pressure (10,000 psi), hot water (190°F) impact tests, a steel plate was placed behind the carton to increase the severity of the test.

The results of the tests were as follows:

1. There was no indication of ignition at any of the test conditions for either Class 1.3 or Class 1.1 propellant.

2. An increase in water temperature improved cutting effectiveness for some propellants but had no effect on propellants having hydroxy-terminated polybutadiene (HTPB) binder systems (Figure 5).

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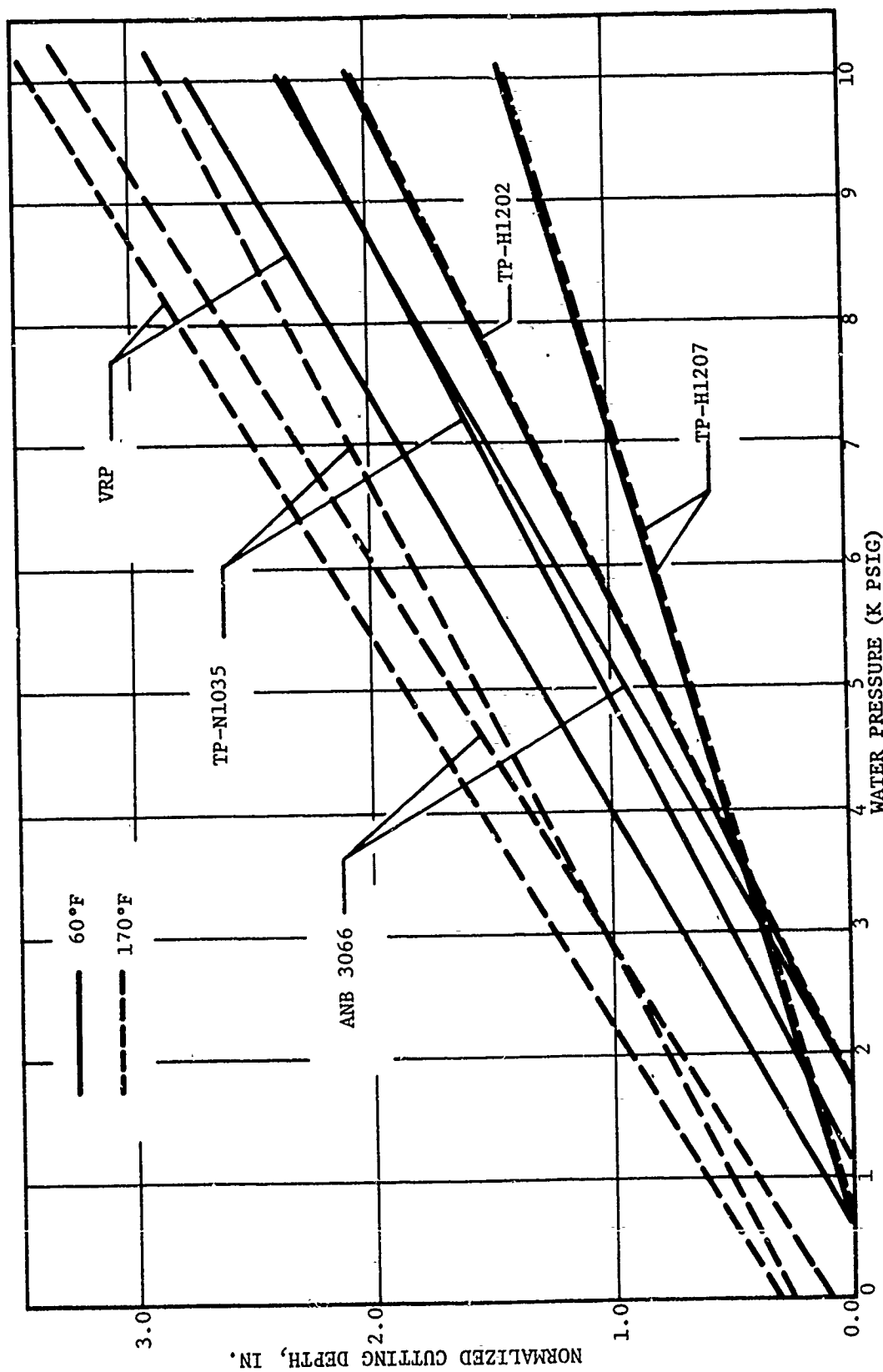
Figure 5. Results of linear multiple regression of sweep cutting rates using the water temperature and pressure as the independent variables (normalized cutting) depth is the calculated cutting depth at a sweep rate of 1.0 in. per sec)

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3. The principal parameter affecting cutting rate was the water pressure. The nozzle diameter also had a measurable effect on cutting rate shown in Figure 6.

4. An indicator of the cutting rate can be deduced from the hardness of the propellant. The softer the propellant, as measured by Shore A hardness or penetrometer measurements the deeper the cutting depth (Figure 7).

Machining. The objectives of the machining tests were (1) to determine whether Class 1.1 propellants could be machined at sufficiently high rates to be economical and (2) to attempt to correlate cutting rates with the hazards testing results and mechanical properties of the propellant.

An attempt was made to measure the temperature of the cutting tool or the propellant during milling operations. These tests, described in more detail in Part II, Appendix D, were not successful. A new cutting tool was designed which greatly improved cutting efficiency over th. previously used tools, and no measurable temperature increase occurred during the cutting tests.

The results indicated that either wet or dry machining is a viable method for removal of propellant from the case. Due to the increased safety which wet machining affords, wet machining must be the preferred method. Limitations for removal of all of the propellant are (1) the eccentricity of the case, (2) protrusion of insulation into the propellant as occurs at the bulb or the flap bondline, and (3) the ability to control the cutting tool to a sufficient tolerance as the length of the shaft is increased.

Hazards analysis methods, described in Part II, Appendix C, exist for analyzing the machining operation and minimizing the possibility of ignition of the propellant during machining.

Burnout Method. The objective of this evaluation was to determine the risk and potential damage to the case which would be expected if the propellant were burned, at reduced pressures, from the case.
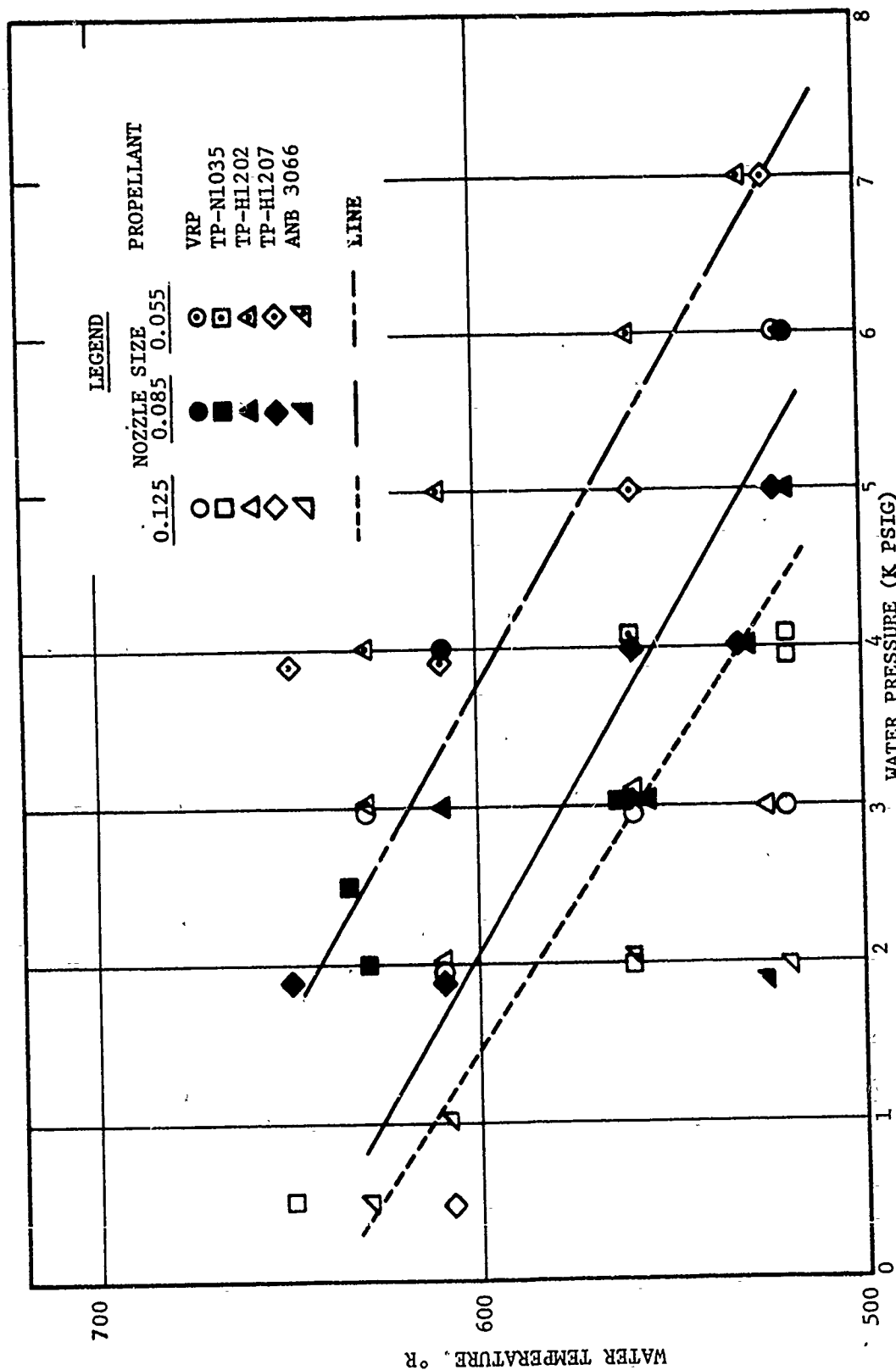
Figure 6. The cut-through point; i.e., the temperature and pressure at which the water jet cut through the 4-in. thick propellant, as a function of the nozzle size

25
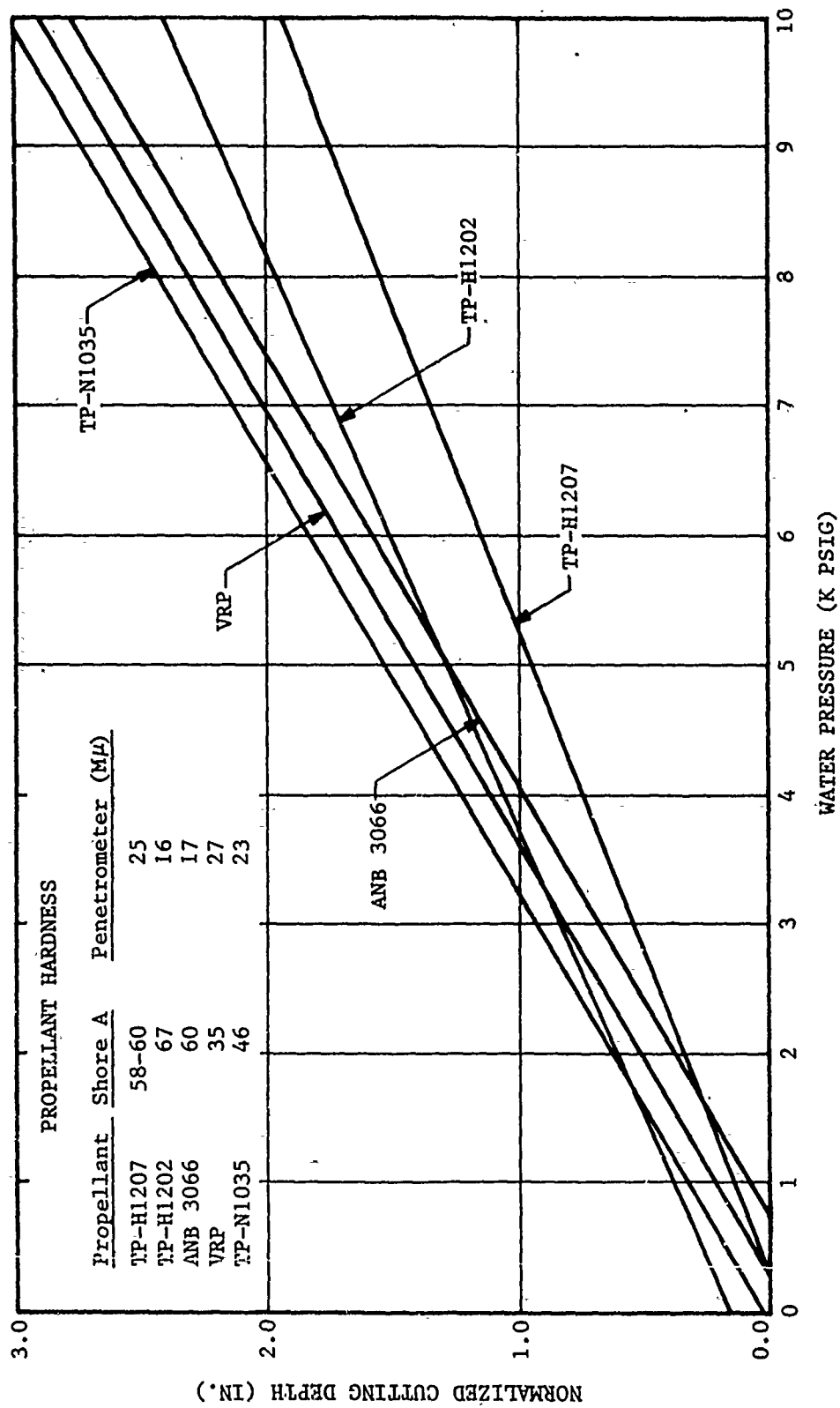
Figure 7. Results of linear regression of the sweep cutting rate as a function of the water pressure. (Normalized cutting depth is the calculated cutting depth at a sweep rate of 1.0 inch per minute)

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A computerized heat-transfer analysis of two motors, the Minuteman III Stage III and the MX Stage I motors, was conducted. In both motors, temperatures at the case/insulation interface were predicted which were unacceptably high. In addition, both motors have experienced accumulations of slag (aluminum oxide) in the motor which greatly increase the char of the insulation and the potential for damage to the case. Reduction of pressure during firing extends the burn time and increases the amount of slag deposition.

It was concluded that the burnout method would not be feasible except in special circumstances, such as when the motor is specifically designed with extra insulation to prohibit damage to the case.

Solvent Degradation of the Propellant. The objectives of the tests of solvents to degrade or desensitize propellants were to determine: (1) whether solvents could facilitate the removal of propellant and improve safety and (2) whether degradation of the propellant with solvents could be integrated into a waste disposal scheme of ingredients recovery. A secondary objective was the determination of the effects of solvents on the other components of the motor. If either of the above objectives were to be applicable, the potential effects of the solvents upon the case materials or the insulation must be identified and, if detrimental, eliminated.

Twenty-eight solvents were selected for testing with six propellants for degradation and/or desensitization. For each propellant, there were several solvents which softened and degraded the propellant and, therefore, could possibly be useful in developing an ingredient recovery scheme for disposal of the waste. One interesting outcome of the test was that the residue remaining after leaching with solvents was as sensitive and, in some cases, more sensitive and, hence, more potentially hazardous than the cured propellant.

2.3.3 The Effect of Solvents on the Propellant/Liner Bond Strength

Tests were conducted on three liner bond systems used in the MX Stage I, Minuteman III Stage III, and First Stage C-4 motors. The C-4 system was selected because of availability and the similarity of the propellant to the MX Stage III propellant. Cyclohexane and ethyl acetate were selected as solvents for this study.

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Bond specimens (90 deg peel and bond-in-tension) were fabricated with insulation that had been exposed to the solvent for 24 hours. After exposure, the insulation was prepared for lining as dictated by the production process. Results of the tests, tabulated in Part II, Appendix C, show that no detrimental effect on the propellant/liner/insulation system would be expected.

2.3.4 The Effect of Solvents on the Insulation

If solvents were used during propellant removal, the insulation would be exposed to solvent and solvent vapors for an extended period. The objective of these tests was to determine whether extended exposure would be detrimental to the insulation.

Two insulations, EPDM-053A and V-45, were selected for testing due to their usage in the MX and Minuteman III motors. Details of the tests performed are given in Part II, Appendix C.

The results of the tests are summarized below:

- 1. Many of the solvents caused the propellant to swell as the solvent was absorbed into the rubber.
- 2. Analysis of the material extracted from the insulation showed that plasticizer (phthalates) was removed by the solvents.
- 3. After removal of the insulation from the solvent, most returned to the original size and visibility showed little or no effect.
- 4. Most of the solvents had no effect on the mechanical properties of the insulation.
- 5. Most of the solvents migrated through the insulation and caused debonding of the case/insulation bond and attacked the resin systems of the case.

None of the tests was designed to show what, if any, long term effects on the aging characteristics of the insulation could be expected. However, the removal of the plasticizer would be expected to be deleterious.

2.3.5 The Effect of Solvents on the Case Materials

Testing of solvents upon the case materials was directed to determine the extent of damage, if any, that might occur during solvent-aided propellant removal operations.

The samples selected for testing included (1) sections cut from cases (2) NOL rings (for short shear beam specimens), (3) rheometric dynamic spectroscopy (RDS) specimens, and (4) 5.75 in. bottles. Details of the tests performed and the results are given in Part II, Appendix C.

The summarized results are:

1. Most of the solvents produced debonding of the insulation from the case in the screening tests conducted with case sections.

2. Most of the solvents significantly reduced the ultimate stress values produced in the short shear beam tests of the Kevlar-49 sample. Only methylene chloride and chloroform significantly affected the Glass S901 samples. Testing of the RDS samples confirmed these results.

3. Solvent migration through the insulation of the 5.75 in. bottles resulted in lower hydroburst pressures or so much damage that testing was not possible.

2.3.6 Insulation Removal

Hydromining. It was concluded from technology reported in literature and from results of the Minuteman III Program that removal of the insulation by hydromining, except the flaps, was a high risk operation with respect to damage to the case. It was determined that the insulation could be removed with 2,500 to 3,000 psia water pressure. Lower pressures did not cut the rubber whereas higher pressures produced excessive damage of the case fibers. With Kelvar cases, severe delamination occurred at all pressures, totally eliminating hydromining as a method of removing insulation from Kevlar cases. The effect of the water temperature on the pressure needed for removal was not determined.

The objective of the Phase III tests was to attempt to characterize conditions which could cause damage to the two insulations of interest on the case structures.

The results tabulated in Part II, Appendix C show that some damage could occur to the insulation at low pressures, 500 to 1,000 psi, if the jet was allowed to dwell too long in one place. It was concluded that hot water, low pressure hydromining could be conducted in a manner which would minimize the potential to damage the insulation. Hydromining was not recommended for insulation removal since pressures which remove the insulation also tend to damage the case fibers (glass cases) or, with Kevlar, water caused severe delamination.

2.3.7 Reinsulation

Since complete removal of the insulation is not recommended, the rein-sulation operations consist of replacement of the flaps and repair of damaged insulation. Both of these operations are performed in new case manufacture and do not constitute new technology.

The objective of the tests performed was to determine how the application of heat and solvents to the insulation would affect the bonding of new rubber to the existing insulation.

The results of the studies, detailed in Part II, Appendix C, show that, when rubber from a fired motor was bonded to new rubber, the new bond was adequate. Failures of the samples generally appeared to be in the ply bond of the new rubber.

The effect of the solvents on the rubber-to-rubber bond strength was varied. With either EPDM-053A or V-45 rubber, some solvents produced higher bond strengths and some lower than the control samples which had not been subjected to solvent exposure. Long term effects of the solvents on the bond strength were not investigated.

2.4 PHASE IV - PROGRAM PLAN DEVELOPMENT

The objective of Phase IV was to outline a follow-on program to demonstrate the salvage techniques selected from Phases I, II, and III using three, government-furnished, Minuteman III Stage III motors.

Because the MX program is an ongoing program which may benefit from the case salvage procedures that have been developed, salvage of MX motors was also included in the follow-on plan.

The program plan, submitted for Phase IV, is included in this report as Part II, Appendix D. This plan outlines a program to salvage three Minuteman III Stage III motors to demonstrate processing techniques for Class 1.3 propellant. Salvage operations for MX Stage I and II motors are also included, in the event that one of these motors should become available. Salvage of one Minuteman II Stage III motor is included to demonstrate salvage techniques required for motors containing Class 1.1 propellant. Alternately, an MX Stage III motor could be used, if available.

3.0 CONCLUSIONS

Based upon the results of the Phase I, II, and III efforts, the following conclusions were made:

1. Existing technology for Class 1.3 propellant removal developed for reclamation of steel cases appears to be applicable to the salvage of composite cases.

2. Salvage of composite cases from solid propellant rocket motors in existing weapons systems is questionable due to unanswered questions regarding the long term aging effects on the case structural system. Also unanswered are the possible long term aging effects which could result due to the salvage operations.

3. Methods of salvage of cases from motors containing Class 1.1 propellant are unproven; however, problems associated with Class 1.1 propellant do not appear to be insurmountable.

4. Salvage of composite cases from motors containing Class 1.3 propellant appears to be cost effective, particularly for large chambers. For motors containing Class 1.1 propellant, the estimated cost savings are marginal even for large motors.

5. The preferred methods for propellant removal are hydromining and machining. Utilization of solvents to soften the propellant is not recommended due to the increased probability to damage the case. The burnout method appears not to be feasible due to predicted high temperatures at the case/insulation interface.

6. Removal of flaps and reinstallation appears feasible and should be planned for.

7. Removal of structural insulation is a high risk operation with high probability of damaging the case.

8. Utilization of solvents to soften the propellant or swell the insulation for removal would probably cause damage to the case by weakening the resin systems.

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9. Reclamation of ingredients from waste propellant appears to be feasible; however, marketability of the reclaimed ingredients is the primary concern.

4.0 RECOMMENDATIONS

1. A follow-on program, outlined in Phase IV, to salvage composite cases should be funded to verify the technology and cost model developed in this program.

2. Motors from existing weapons systems should be included to include a determination of the effect of the salvage operation on long term aging.

3. Salvage of at least one motor containing Class 1.1 propellant should be planned to provide a better data base for predicting salvage costs and to confirm recommended operational procedures.

4. For salvage of motors containing Class 1.1 propellants, remote operations and utilization of high-hazard, expendable facilities and tooling are recommended.

5. Utilization of organic solvents for propellant or insulation removal should be minimized due to the deleterious effects the solvents produce in the case resin systems.

APPENDIX A
PHASE I INTERIM REPORT
TECHNICAL ASSESSMENT

R&D STATUS REPORT

DEVELOPMENT OF
COMPOSITE CASE SALVAGE
PROCEDURES

SUBMITTED TO

DEPARTMENT OF THE AIR FORCE
HEADQUARTERS, AIR FORCE FLIGHT TEST CENTER
DIRECTORATE OF CONTRACTING (PKRA)
EDWARDS AFB, CA   93523

In Accordance With
Contract F04611-81-C-0001
CDRL No. 1

January 1981

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SUMMARY

This is the first report on the program from the Development of Composite Case Salvage Procedures, AFRPL Contract F04611-81-C-0001. The major portion of Phase I, Technical Assessment, has been completed. A continuing low level effort will proceed through Phases II and III. The overall program is outlined to develop safe, cost effective procedures to remove Class 1.1 and Class 1.3 propellants from solid rocket motors. The program includes a 15-month technical effort followed by a 3 month final report cycle and is divided into the following four phases, each having a separate objective:

      I.   Technology Assessment
     II.   Feasibility and Cost Studies
   III.   Laboratory Studies
    IV.   Demonstration Program Plan

Phase I was initiated with a review of literature. These reviews included:

1. The review of literature for propellant grain degradation using solvents and/or reagents.
2. The current state-of-the-art of the physical removal techniques.
3. Waste propellant disposal methods.
4. Insulation removal and replacement techniques.
5. The effects that propellant removal and insulation removal and replacement may have upon the composite rocket motor structure system.

Propellant Grain Degradation Using Solvents and/or Reagents

The literature review on methods to degrade the propellant polymer systems indicated a number of solvents and reagents

that could be used to destroy the structural capability of the various binder systems. Many of the candidates require further study to determine the degradation rates, the permeability of the solvent and chemicals into the propellant grains and the compatibility of the solvents or reagents with the propellants. Since propellant grain degradation by chemicals is a unique process, several areas have since been found to hold some promise and further '.. :ature research is required. Investigation in these areas is continuing.

Physical Method of Propellant Removal Techniques

In the area of physically removing the propellant from the rocket motor cases, the state-of-the-art that is the most advanced is the hydromining and the mechanical cutting removal techniques. Several facilities and plantsites are found to exercise this capability. Thiokol Corporation probably has the largest hydromining facilities for propellant removal. Hercules Incorporated is most knowledgeable in machining propellants that contain liquid explosives such as nitroglycerin. Aerojet. has limited facilities for hydromining and is currently developing new hydromining facilities. The NOS has conducted many studies and has built pilot plants for hydromining rocket motor propellants. NOS sublet a contract to Thiokol to help them design an advanced hydromining facility. The only reference for hydromining double base rocket motor propellant stems from an English report, the IMI Limited, Summerfield Research Station at Kiddermunster, Great Britain, wherein a fire caused by the high pressure jet action on nitroglycerin pockets in rocket motor grains destroyed their facility. Additional work for hydromining propellants that contain liquid explosives needs to be thoroughly examined.

Waste Propellant Disposal Techniques

The propellant disposal techniques, as presently practiced throughout industry for explosive Class 1.1 and 1.3 materials, is limited to open air burning. Less than 1% of all of the

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hazardous explosive materials are disposed of with other proc-
esses. The Army plant at Radford, Virginia, operated by Her-
cules and the Army plant at Tooele Ordnance Depot each have
rotary kiln explosive and propellant disposal incinerators.
The unit at Radford is a firebrick-ceramic lined rotary kiln
while the unit at Tooele Ordnance Depot is a 3-in. thick steel
rotary kiln, sometimes referred to as a popping furnace. The
Army Depot at Dover (ARRADCOM) has experimented with fluidized
bed incinerators and presently a contract is being considered at
the Tooele Ordnance Depot to further evaluate fluidized bed
incineration of propellants and explosives. A third experi-
mental method of disposing of propellants and explosives is the
NOS Indian Head wet air oxidation method where high pressure,
high temperature steam is used to decompose the organic sys-
tems.

Both Thiokol/Wasatch and the Army plant at Radford are
entertaining the idea of selling their waste explosive mate-
rials to blasting companies. Radford finds an interest through
blasting companies who supply blasting compounds to the coal
mines. Thiokol is finding an interest from suppliers who sup-
ply blasting compounds to the mining industry.

Insulation Removal and Replacement Technique

Insulation removal and replacement technology has been
limited at the present time to repair techniques required dur-
ing the fabrication of insulated cases. Several large motors,
156 in. in diameter, have had massive amounts of insulation
removed due to unbonded conditions and replaced. The motors
fired successfully. The technology does exist for removing the
insulation from fiber composite rocket motor cases and replac-
ing it with newly fabricated insulation. Work is being done in
this area at the present time at Thiokol/Wasatch in conjunction
with the Thiokol Corporation analysis of Minuteman Stage III
retrofit potentials.

Case Structural Considerations

The composite rocket motor case structural system has been extensively studied. Much information is available about the different composite systems, their effects by moisture, their effect by repeated loading and their design limitations. The literature searches provide data regarding methods of analyzing the systems to account for exposure and multiple loading.

CONCLUSIONS AND RECOMMENDATIONS

Propellant Grain Degradation Using Solvents and/or Reagents

Propellant removal needs to be further investigated to determine the rates of removal, compatibility, and permeability of the systems. The continuing technology assessment effort will be concentrated in this area.

Physical Method of Propellant Removal Techniques

The physical methods of propellant removal, hydromining and machining, are at an advanced state-of-the-art position. New methods to be considered at the present time include the high temperature water to remove the propellant by hydromining and high temperature water to remove certain types of rubber insulation and to clean the case compositions. In mechanical propellant cutting the major limitations at present are safety and machine design. Other unique methods for propellant re-. moval such as burning the propellant from the case have been used with limited success in the past.

Further studies in the physical methods for propellant removal will be conducted in Phase III and be limited to the high temperature water removal process, the hydromining evaluations of nitroglycerin type (Class 1.1) propellants and paper studies with regard to the removal of propellant by burning.

Waste Propellant Disposal Techniques

The propellant disposal state-of-the-art technology is
limited to open air burning. The only data available for other
methods of disposal are in the experimental and pilot plant
stage. Cost evaluations used from these experimental methods
of disposal will be pure estimates based upon knowledgeable
people's evaluation. It is recommended that studies above and
beyond the scope of this program be initiated to provide
feasible alternatives to open air burning.

Insulation Removal and Replacement Techniques

An application of current repair technology needs to be
extrapolated to rocket motor salvage and reconstruction meth-
ods. Additional assessment in this area will be continued.

Case Structural Considerations

Composite case structural systems are well defined both
from an analytical and physical point of view. Methods of
analyzing the effects that propellant removal technologies and
insulation removal technologies have upon the case structural
system are sufficiently developed that design criteria and
testing criteria can presently be outlined. Feasibility and
cost effective propellant removal techniques need to be re-
viewed and tested to determine the effects on the composite
structural system. The effort will be accomplished in Phase
III.

1.0 INTRODUCTION

The objective of the program for the Development of Composite Case Salvage Procedures, AFRPL Contract F04611-81-C-0001 is to develop safe, cost-effective process technologies for salvaging and recycling composite cases from solid propellant rocket motors. The motors evaluated in this program study range from 4,000 lbm to 200,000 lbm and are loaded with either Class 1.1 or 1.3 propellant.

The program includes a 15-month technical effort followed by a 3-month final report cycle. The program is divided into the following four phases, each having a separate objective:

    I.   Technology Assessment

    II. Feasibility and Cost Studies

  III. Laboratory Studies

  IV. Demonstration Program Plan

This report presents the status of the Phase I Technology Assessment. The details of the literature search are presented in the five sections described below.

Section

2.0 Propellant Grain Degradation Using Solvents and/or Reagents

3.0 Physical Method of Propellant Removal Techniques

4.0 Waste Propellant Disposal Methods

5.0 Insulation Removal and Replacement Techniques

6.0 Case Structural Considerations

In each section, the technology reviewed is presented with abstracts based upon the procedures and systems considered applicable to the development of composite case salvage procedures. Each section also contains a summary of abstracts with applicable conclusions and recommendations.

2.0 PROPELLANT GRAIN DEGRADATION USING SOLVENTS AND/OR REAGENTS

Although degradation of propellant grains using solvents and/or chemical reagents has not been studied in great detail, ample information is present in the literature to substantiate the feasibility of this approach and to suggest efficient lines of attack. Solvent swelling and extraction, hydrolysis using inorganic acids or inorganic or tertiary amine bases, and transesterification all appear promising candidates for laboratory studies. The major unknown elements remaining to be defined include: (1) the rates of degradation of propellant grains, (2) the permeability of propellant grains to solvents/reagents, and (3) the compatibility of grain constituents with the solvents/reagents employed under the conditions used for removal.

Solvent/reagent methods for removal of propellant grains appear promising and should be thoroughly investigated. Literature and technical assessment work will be continued, particularly in areas where the chemistry shows additional promise towards degrading polymer structures. The literature search conducted in this area is summarized in Tables 2-1 and 2-2.

TABLE 2-1

SUMMARY OF LITERATURE SEARCH ON PROPELLANT GRAIN DEGRADATION
USING SOLVENTS AND/OR REAGENTS

Literature Search Designation	Number of Initial Leads	Number of Apparently Relevant References	Number of Directly Applicable References
Topical Survey of Chemical Abstracts and Current Journals	186	76	10
LMSC-Chemical Abstracts (A computer based search using fixed identifiers)	579	116	7
LMSC (NTIS-64-80) (A computer based search using fixed identifiers)	353	60	1

TABLE 2-2

IDENTIFIERS USED IN COMPUTER ASSISTED
LITERATURE SEARCHES

A.   Matrix used for chemical abstracts search

Polymer		Urethane		Hydrolysis
Rubber	plus	Polyester	plus	Degradation
Elastomer		Polyether		Cleavage
				Recycling
				Reclamation
				Solvoly--

B.   For LMSC search

Salvage		Rubber
Reclamation		Elastomers
Degradation	plus	Solid Propellants
Machining		Nitroglycerin
Disposal		Explosive Materials

2.1 ABSTRACTS AND BIBLIOGRAPHY OF DIRECTLY APPLICABLE
    REFERENCES

Comparison of Some Soft Unplasticized Cast Polyurethane
Rubbers,
 G. B. Guise and G. C. Smith, J. Macromol. Sci., Chem.
1980, A14(2), 213-32. (Eng)

>   Abstract: Hydrolysis, solvent swelling and
>   properties are discussed as functions
>   of composition, fillers, etc.

Magnetic Resonance Studies of Epoxy Resins and
Polyurethanes,
>   I. M. Brown, et al., Report, 1979, MDC-Q0673;
>   Order No. AD-A073590, 110 pp. (Eng)

>   Abstract: Proton NMR was used to investigate
>   hydrolysis. Poly(ester-urethane)
>   underwent hydrolysis and
>   "catastrophically depolymerized from
>   rubber solids to viscous liquids."

Kinetics of Hydrolytic Aging of Polyester Urethane
Elastomers,
>   D. W. Brown, R. G. Lowry and L. E. Smith,
>   Macromolecules, 1980, 13(2) 248-52. (Eng)

>   Abstract: Results from acid-catalysis.
>   Equations are given as are rates
>   and activation energy.

Recycling of Thermoset Polyurethane Elastomers,
>   H. Ulrich, et al., J. Elastomers Plast., 1979,
>   11, 208-12.

>   Abstract: Heating polymers with dipropylene
>   glycol gave degradation to homogen-
>   eous polyols.

The stability of Elastic Integral Polyurethane
Foams Toward Some Selected Organic Solvents,
>   H. J. Oder and B. Naber, Plaste Kautsch., 1980,
>   27(2), 88-90 (Ger)

>   Abstract   Polyester-based polyurethane foams
>   are resistant to gasoline, diesel
>   fuel, Cl_3CF, MeOH, EtOH and i-
>   PrOH, but not to chlorinated
>   hydrocarbons, acetone, DMSO and DMF

Biodegradation at Diisocyanate-Extended
Copolymers, M. M. Bitritto, et al., J. Appl.
Polym. Sci. Appl. Polym. Symp., 1979, 35,
405-14 (Eng)

Abstract: Aspergillus Nigar gave degradation.

Reclamation of Urethane Polymers, K. Hara and H.
Higaki (Asahi Chemical Industry Co. Ltd) Jpn.
Kohai Tokkyo Koho, 79,117,580, 12 Sep 1979, 7
pp.

Abstract: Polyether polyurethanes are
decomposed in mixtures of alkali
metal compounds, H_2O or
solvents having active H groups,
and dialkyl ethers of glycols,
sulfolane, DMSO, 4-methyl-1,3-
dioxolan-2-one, and/or Me_2NCN.
Thus, when 20 g or rigid
polyether/polyurethane foam was
heated in 50 g 95/5/100 glycerol-
KOH-DMSO at 3°/min, the initial
decomposition temperature was 133°F
and the decomposition time at 153°F
was 100 minutes.

Polyol-Containing Liquids from Polyurethane Wastes,
G. Bauer, Ger, Offen., 2,759,054, 12 Jul
1979, 36 pp.

Abstract: Alkali and alkaline earth metals or
compounds were more effective
catalysts than alkanol amines.
NaOAe in diethylene glycol was
effective. Temperatures were high
(approximately 200°F).

Hydrolysis of Urethane Foams (Ford Motor Company),
Jpn., Kohai Tokkyo Koho, 79 70,377, 6 Jun
1979, U. S. Appl. 843,777, 20 Oct 1977, 4 pp.

Abstract: Hydrolysis of urethane foams by
superheated steam is accelerated by
alkali metal hydrohides. Thus, a
urethane foam repregnated with 0.1
phr NaOH (as aq solu), treated with
steam gave 94.3% degn versus 64.9%
without impregnation.

Hydrolysis of Polyurethane Foams, L. R. Mahoney (Ford
    Motor Co.,), Belg. 869,046, 16 Nov 1978.

Abstract   Scraps are heated at 185°/0.5-1.5
                atm in presence of H_2O and
                0.001-0.2 and NH_3/mol H_2O
                to hydrolysis

Solvolytic Degradation of Pyrotechnic Materials
    Containing Crosslinked Polymers, A. S. Tompa,
    et al., U.S. 4,098,627, 15 Dec 1976 (to U.S.
    Dept. of Navy).

Abstract: Covers propellant disposal,
                solvolytic recovery of
                constituents, including aluminum
                and AP.

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    Based Adhesives, W. Fischer, et al.,
    Adhesion, 1978, 22(5), 138-42 (Ger)

Abstract: Pending
Recovery of Polyurethane Prepolymer and Amine Salt,
    D. F. Lohr and E. L. Kay, U.S. 4,035,314, 2
    June 1975 (to Firestone Tire and Rubber Company).

Abstract: Pending

Response of Some Polyurethanes to Humid Environments,
    L. B. Jensen and H. P. Marshall, Compat.
    Propellants, Explos. Pyrotechnics Plast.
    Addit., Conf., 1975, III-E, 12 pp.

Abstract: Pending (Includes Kinetics of
                hydrolysis of polyurethane
                elastomers.)

Solvolytic Degradation of Polymeric Propellant
    Binders, M. S. Kaufman, et al., U.S. NTIS
    AD rep., 1975, AD-A017235, 30 pp.

Abstract: Pending (Covers solvolysis of
                polyester and polyurethane binders,
                waste disposal, catalyst effects,
                etc.)

Degradation Reaction of Urethane Polymers, I.
Transesterification of Polyether-Based
Polyurethane Foam, Y. Numata, et al., Nippon
Gomu Kyokaishi, 1974, 47(12), 839-45 (Japanese).

Abstract: Pending (Reports polyol recovery
from transesterification of
polyurethanes.)

Degradation of Polyurethane Foam, H. Okanoto and K.
Fukada, Japan (73) 08357, 1973 (Japanese).

Abstract: Pending (Reports hydrolysis using
sulfuric acid and polyether
recovery.)

Breakdown of Urethane Elastomers Under the Action
of the Epoxide Tertiary Amine System,
Antipova, V. F., et al., Kauch. Rezina, 1972,
31(1), 14-16 (Russian).

Abstract: Pending

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Comprehensive View of the Combustion Models of Composite Solid Propellants, K. Kishore, AIAA Journal, 1979, 17(11), 1216-24. (A review with 65 references)

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Characteristic Effects in the Reaction Kinetics of Polymeric Reagents, H. Morawetz, Pure Appl. Chem., 1979, 51(12), 2309-11 (Eng) (A review with 35 references)

Developments in Polyurethane, Vol 1, J. Buist, Ed. (Applied Science Publishers, Ltd.) 1978, 280 pp

The Synthesis and Properties of Polyurethane Resins, Vol 2 (1973- October, 1979), D. Cavagnaro, Report 1979, Order No. PB80-800477, 270 pp. (Eng) (A bibliographic review with 293 references) (Avail NITS)

Developments in Polyurethane Elastomers, R. P. Redman, Dev Polyurethane, 1978, 1,33-76, (Eng) (A review with 143 references)

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Permeability of Heterogeneous Gels, N. Weiss, et al., J. Polymer Science, Polymer Physics Ed., 1979 17(12) 2229-40.

IT-M54-45-9   IHMR-71-162 "Evaluation and
               Characterization of Binder
               Constituents," Quarterly Progress
               Report, D. M. French and M. Graff,
               Indian Head, Maryland

               Abstract: Twenty four of newer
               butadiene liquid polymers were
               characterized with respect to a number
               of properties

               Not applicable except 71 costs of
               materials are cited

IT-M54-53-34   IHTR 273 "Thin-Layer Chromatography -
               Method Applicable to the Separation and
               Identification of Complex Organic
               Compounds Present in Double-Base
               Propellants," Naval Ordnance Station,
               Indian Head, Maryland, August 1968

Comments: Solvents used may be useful in
               degradation of propellant. Relative
               retention rates in respective solvent
               systems are presented

3.0 PHYSICAL METHOD OF PROPELLANT REMOVAL

The current and ongoing literature search includes the hydromining of propellants, mechanical cutting of propellants, and other techniques that are available in the industry today.

To date the literature search has not revealed any new or unusual methods for propellant removal. Most of the pertinent literature concerning hydromining of propellant has been generated inhouse by Thiokol. The basic methods of physical propellant removal to be evaluated for Class 1.1 and 1.3 propellants are: hydromining and machining.

3.1 HYDROMINING

During the 1960s, Thiokol/Wasatch built and developed the nation's leading solid rocket motor case reclamation facility (Figure 3-1). This unit used the hydromining technique, whereby high pressure water jets carve out propellant pieces until the entire grain was removed.* A second, but much smaller hydromining facility was later installed by Thiokol/Elkton in Maryland. Thiokol's experience is summarized in Table 3-1. Significantly, this table shows that Thiokol's experience falls into two major categories: reclaiming steel cases and reclaiming large composite cases. In either situation the propellant removed is a composite formulation. Both insulator and liner were completely removed from steel cases while insulation was left intact in composite cases.

The economic incentive for development of Thiokol/Wasatch Division case reclamation facility was initially provided by Minuteman first stage motor cases. Because of this, the facility was designed to handle large motors, although it was readily adapted to recover the relatively smaller Bomarc motors.

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*McQueen, H. F., and Ladd, J. C., Rocket Motor Case Reclamation, Thiokol/Wasatch Division, May, 1964.
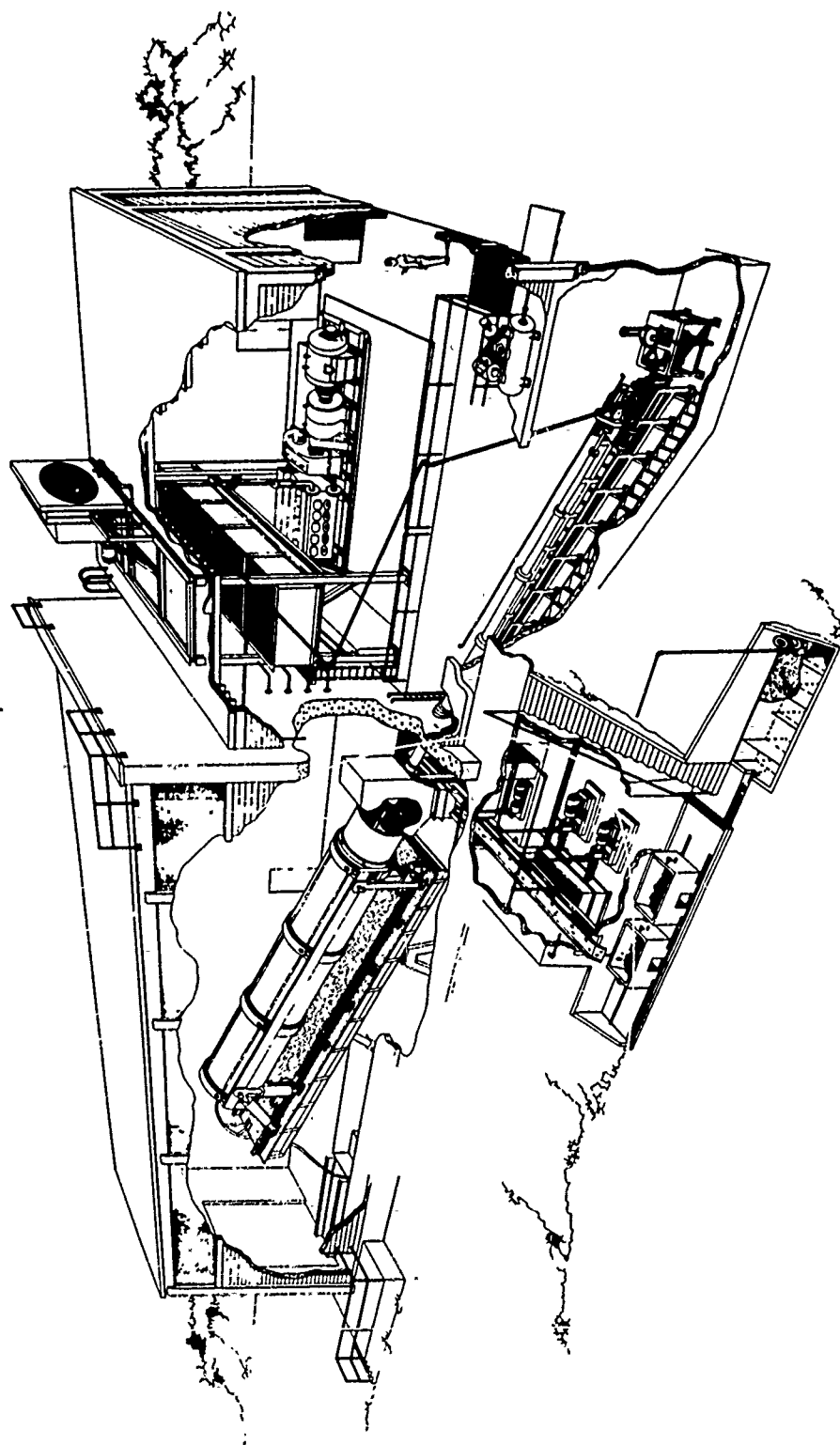
Figure 3-1. Wasatch Division Case Reclamation Facility

TABLE 3-1

HYDROMINING SUMMARY OF SOLID ROCKET MOTORS RECLAIMED BY THIOKOL

Motor	Number Reclaimed	Case Material	Case Size	Propellant Weight (lb)	Propellant Type	Comments
Minuteman, 1st Stage	330	Steel	21 ft x 65.5 in. dia	44,000	Composite	
Bomarc	22	Steel	10 ft x 35 in. dia	3,000	Composite	
Genie	2,321	Steel	54 in. x 14.9 in. dia	320	Composite	
HARM	1	Steel	5.5 ft x 10 in. dia	260	Composite	
Castor II	7	Steel	19.6 ft x 31 in. dia	8,200	Composite	
Castor IV	16	Steel	30 ft x 40 in. dia	20,664	Composite	
Scout (Algol)	1	Steel	26.5 ft x 45 in. dia	28,000	Composite	
Subroc	320	Steel	--	2,000	Composite	Done at Thiokol/ Elkton, Maryland
SRAM	5	Steel	100 in. x 17.6 in. dia	1,453	Composite	Portable equipment used; first motor igniter
Poseidon, 1st Stage	1	Glass	14.75 ft x 84 in. dia	38,000	Composite	No significant damage to insulation; low pressure steam used for final cleanout
Trident-I (C-4), 1st Stage	1	Kevlar	14.75 ft x 84 in. dia	41,500	Inert }	No damage to insula- tion; propellant was
Trident-I (C-4), 2nd Stage	1	Kevlar	8 ft x 84 in. dia	18,450	Inert }	inert
Pershing	16	Steel			Composite	Economical evaluation completed

Additional small motor case recovery hydromining capability was developed at the Elkton, Maryland facility, which was used to remove propellant from Subroc motors.

Thiokol's involvement in the Genie Program included complete redevelopment of the propellant system. Initially, the Aerojet propellant had a 27 mo shelf life, which Thiokol replaced with a propellant having a 12 yr shelf life and wider thermal limits. Consequently, more than 2,000 Genie motors have been reclaimed at Thiokol/Wasatch by hydromining.

Reclamation of Castor II and Castor IV from Elkton Division and the Scout (Algol) UTC/CSO motor is indicative of the Wasatch Division's ability to salvage motors that are not produced inhouse. Reclamation of these motors necessitated thorough propellant hazard analysis prior to hydromining to insure safety.

HARM is a new development program, and the only motor that has been reclaimed had a casting defect. Moreover, HARM is unique in that it has a very hard asbestos filled phenolic insulator that cannot be removed by a 6,000 psi water jet. Thiokol/Wasatch developed a special baking procedure to remove HARM insulation from the one motor that was hydromined and other motors that have been fired.

Hydromining of SRAM motors became necessary when Thiokol won the redevelopment contract for this motor. At the time, no motor cases were available and Thiokol had no data for hydromining these motors. It was decided to attempt reclamation of five motors. Prior experience of other contractors indicated a high incidence of motor ignition during washout, possibly resulting from the presence of a live igniter buried in the propellant. An isolated hydromining system was set up away from the M-115 hydromining facility to avoid damage in case of a fire. The first motor ignited and the case was damaged.

A cause of ignition was postulated as water friction acting on a friction sensitive propellant and, with the experience gained in the first attempt, the remaining four motors were successfully salvaged.

Extending the technology for propellant removal from steel cases to filament wound composite motor cases without damaging insulation or case was highly desirable. Two retrofit programs that could benefit from this development are the reclamation of Poseidon First Stage and Minuteman Third Stage motors. In 1971 Thiokol management decided to attempt salvaging a reject Poseidon First Stage motor using modified hydromining techniques. This experiment proved quite successful. Using procedures developed on Minuteman First Stage, high pressure water was used to wash out most of propellant. As the case wall was approached, fan shaped nozzles and lower water pressures, as well as faster nozzle rotation speeds, were used to reduce the possibility of insulation or case damage. Finally, low pressure steam was applied for up to 64 hr, which caused propellant softening to a depth of approximately 3/4 inch. The soft propellant was easily removed by low pressure water. This approach allowed removal of an eccentricity (excess propellant on one side of the motor) without damage to insulation or case on the other side. Results of this test showed the case to be totally undamaged while the insulation was undamaged except for several small areas, which were easily reparable. Effects of steam on the case or on subsequent bonds between insulation, new liner or new propellant were not evaluated.

Similar experiments using Trident-I (C-4) first and second stage motors (Kevlar cases) were conducted in 1977. Both units were designed as ground test motors and were cast with an inert XLDB (crosslinked, double base) propellant without nitroglycerin that represented the Trident-I (C-4) VRP propellant. In each situation, unacceptable cures necessitated salvaging

the cases. Techniques developed for the Poseidon First Stage allowed all propellant to be successfully removed without damage to cases or insulators and the motors were recast with the inert propellant. Figures 3-2 thru 3-4 illustrate this process for the Trident-I (C-4) second stage motor.

Tilt table mounting and positioning of the programmable water lance are shown in Figure 3-2. A low pressure water hose is mounted to the case forward end to sweep out debris. Figure 3-3 shows bulk propellant removal and the effect of water jets on propellant, while Figure 3-4 shows the finished case with all propellant removed.

In recent years Aerojet-General Corporation has commissioned a hydromining facility, which is now being used to salvage Minuteman second stage motors. These motors have a titanium case and are loaded with ANB-3066 propellant. Aerojet has also salvaged at least one Minuteman third stage motor having a fiberglass case and an ANB-3066 propellant grain. Hydromining was used to remove the bulk propellant, followed by water and/or steam soak to remove propellant next to the insulator. Minuteman second and third stage motors which were loaded with ANB-3066 have a compatible liner which is easily degraded with water. Clearly, this work confirms that the application of techniques developed at Thiokol can be successfully applied to other motors.

Thiokol and other USA propulsion contractors have not hydromined double base or crosslinked double base propellants from rocket motors. Obvious problems associated with this operation involve increased ignition probability and waste water handling. However, work done in this area by Summerfield Research Station, Kidderminster, Great Britain is worth reviewing.*

*Bingham, J. F., et al., Removal of CDB Propellant From Case Bonded Rocket Motors by High Pressure Water Jet, IMI Ltd, Summerfield Research Station, Kidderminster, GB
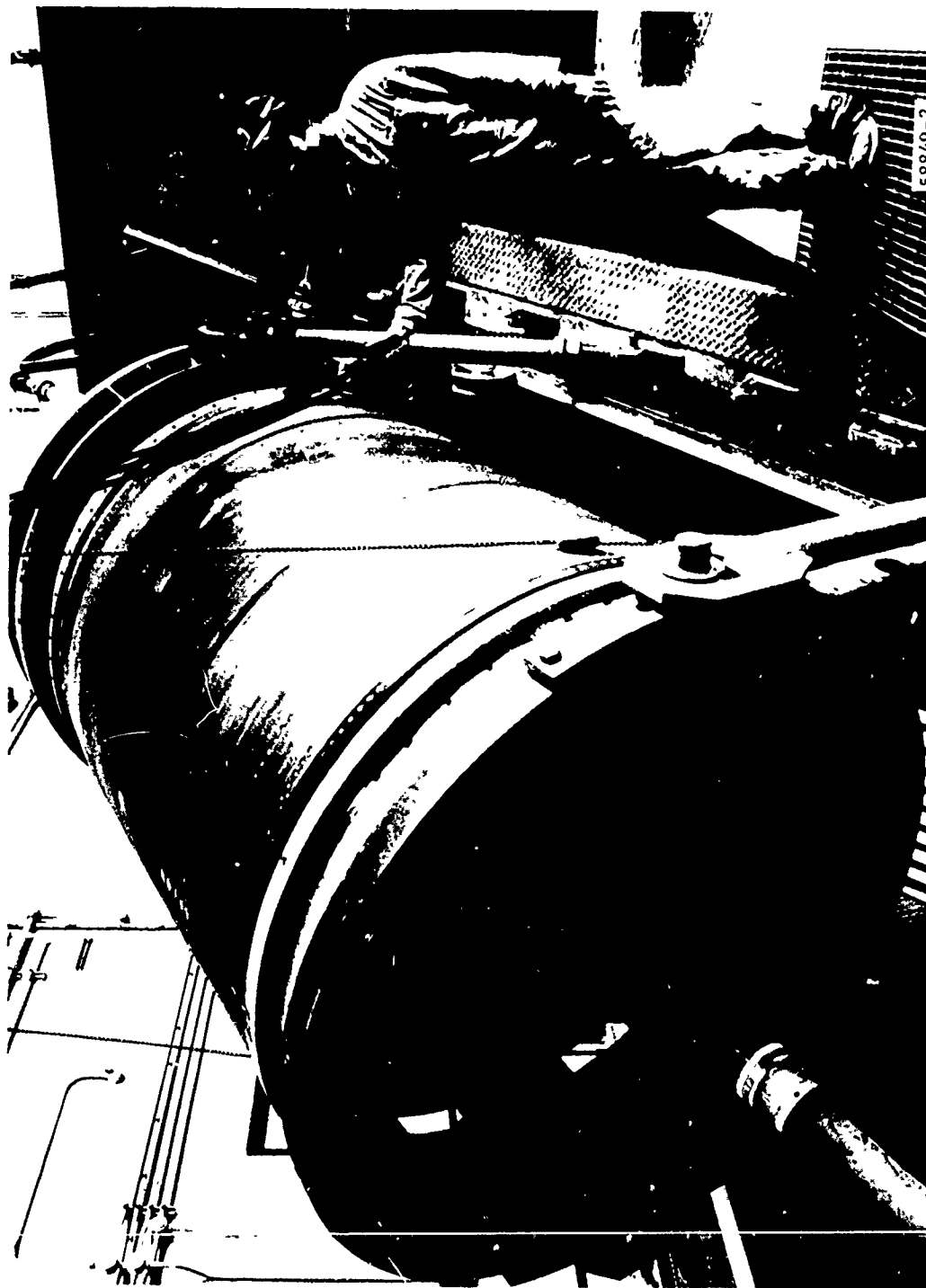
Figure 3-2. Trident-I (C-4) Second Stage Motor Mounted on Tilt Table

58849-2

58849-1
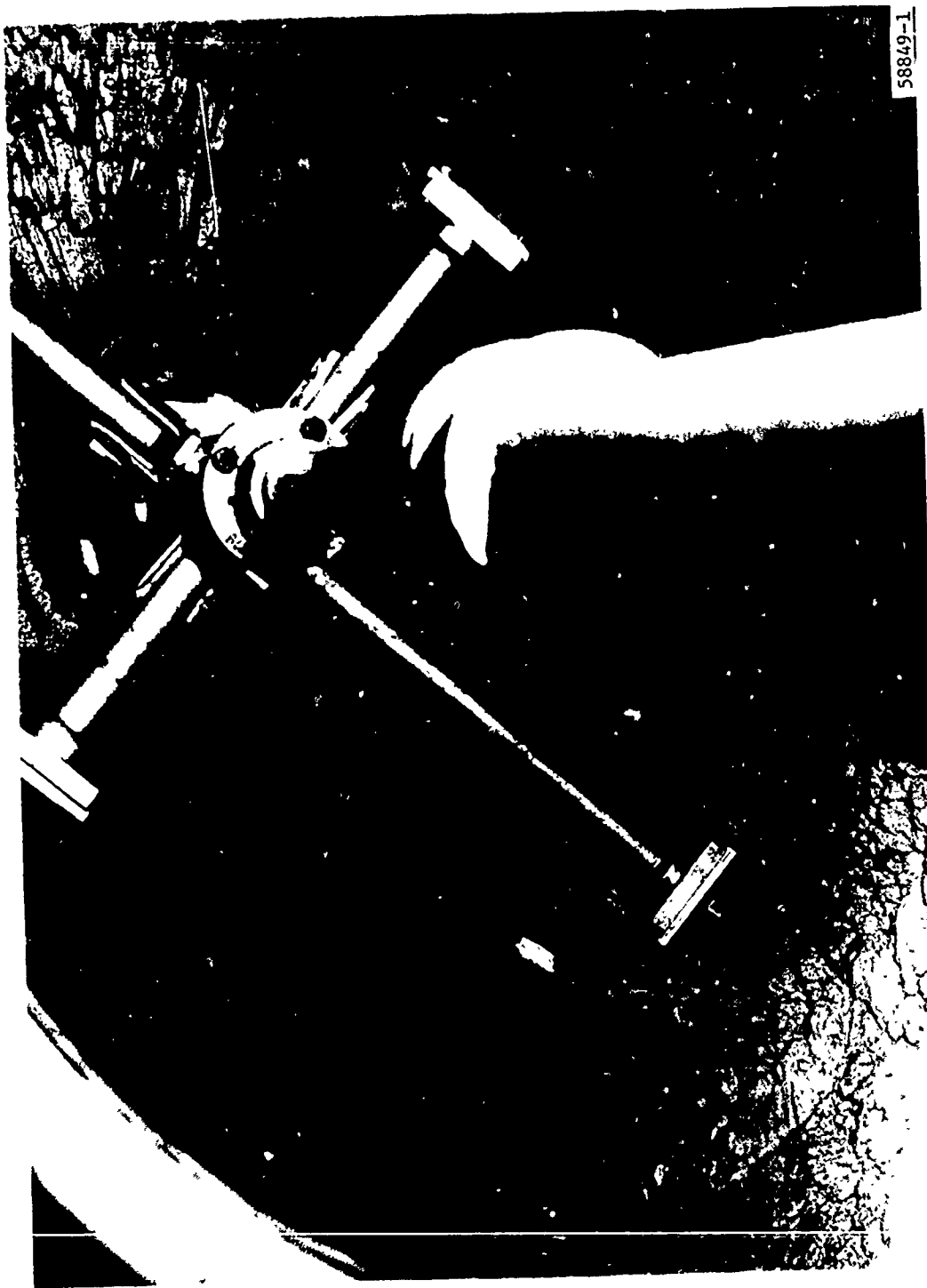
Figure 3-3. Bulk Propellant Removal
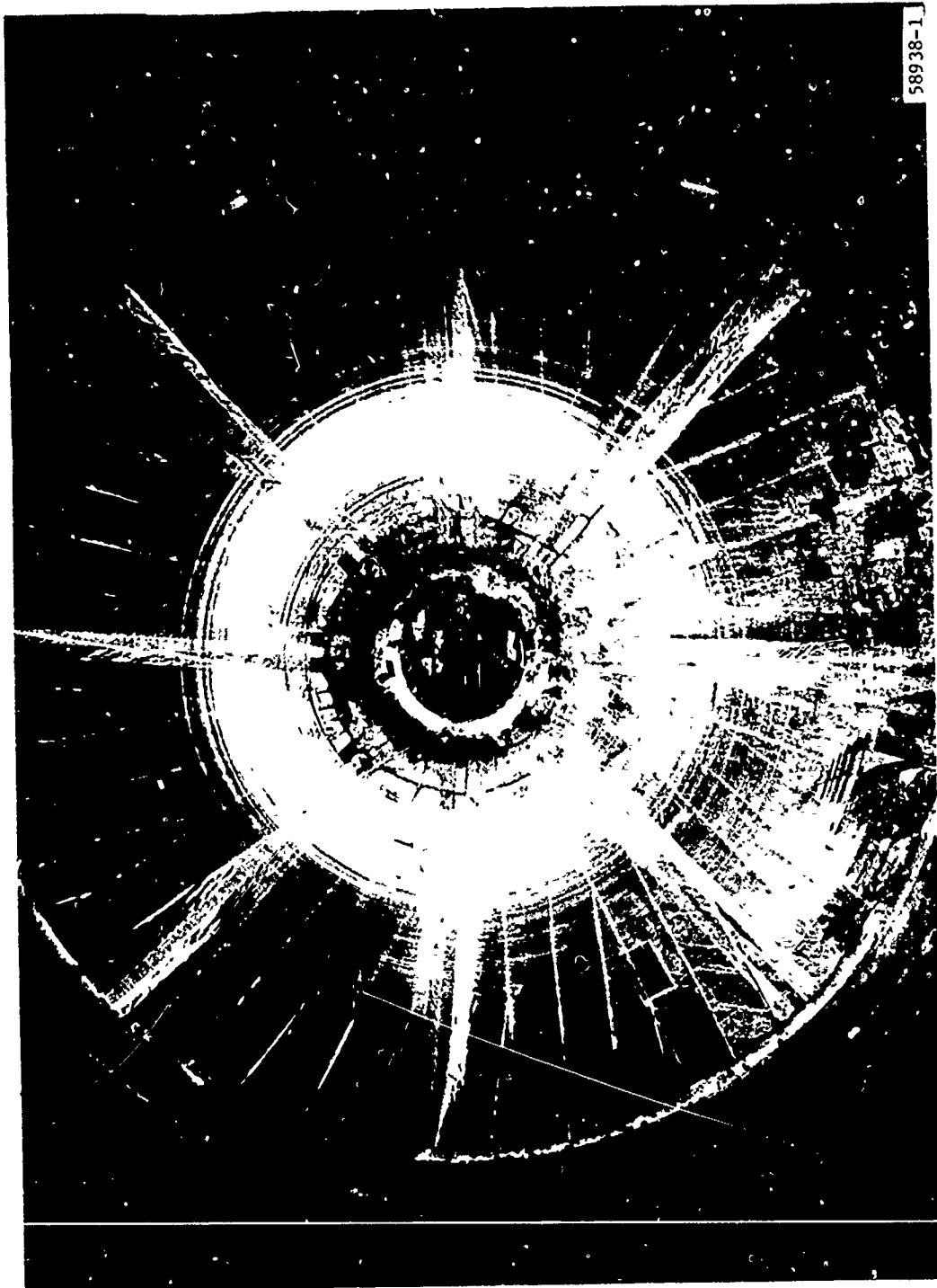
Figure 3-4. Salvaged Trident-I (C-4) Second Stage Motor

58938-1

A-29

This facility used hydromining (10,000 psi noncavitating water jet) to remove case bonded cast-double-base propellant and insulation from steel motor cases. In 1.5 yr of operation, 250,000 lb propellant has been removed. The problem of nitroglycerin in effluent water was addressed by using sodium hydroxide hydrolysis, then hydrochloric acid neutralization prior to discharging the water. Unfortunately, this facility was seriously damaged in July 1977 when a motor ignited. The subsequent fire investigation suggested that voids filled with casting fluid (nitroglycerin) could have contributed to motor ignition when struck by high pressure water.

3.2 MECHANICAL CUTTING (MACHINING)

Mechanical propellant cutting or machining has been in common use for over 20 years. Standard procedures include the use of types of milling machines or lathes, and many solid rocket motor contractors such as Hercules, Aerojet, UTC and Atlantic Research have worked in this area. Mechanical propellant cutting is a proven technology which snould be considered as a propellant removal method for reclaiming rocket motor cases.

Propellant machining has had two primary applications. The first involves repairing defects such as cracks or separations wherein the defective region is cut out and new propellant cast into the cavity. The second is a means of propellant grain forming by machining overcast motor grains. Thiokol has extensive experience in both applications (Table 3-2). The Elkton Division (Maryland) specialized in cutting grain configurations into space motors by using a vertical turret lathe to dry machine composite propellant. The Wasatch Division (Utah) has repaired and shaped the propellant grain on large motors that fit the scope of this program as well as on small motors.

TABLE 3-2

MECHANICAL CUTTING SUMMARY OF SOLID PROPELLANT ROCKET MOTORS REWORKED BY THIOKOL

Program (Motor)	Number of Units Machined	Average Pounds Propellant Removed/Unit	Total Pounds of Propellant Removed	Case Type	Propellant Type	Propellant Class	Comments
120 In. Motor TU-465.01	1	10,300	10,300	Steel	PBAN	1.3	Machining done dry. Purpose was grain repair
156 In. Motor TU-312L.02	1	23,400	23,400	Glass	PBAN	1.3	
Minuteman							
First Stage	2,973	400	1,189,000	Steel	PBAN	1.3	Machining done dry. Purpose was to cut back overcast grain
Second Stage	*	*	*	Titanium	CTPB	1.1	Dry machining
Trident-I (C-4)							
First Stage	203	210	42,630	Kevlar	CTPB	1.3	Wet machining
Second Stage	**	**	**	Kevlar	XLDB	1.1	Wet machining
Genie	6,679	8.5	56,800	Steel	CTPB	1.3	Dry machining

*Most probably the same as Minuteman First Stage; however, Aerojet Corporation data is not directly available
**Similar to Trident-I (C-4) First Stage

A-31

Wasatch has had two particularly interesting and germane defect repair projects which provided experience for this program in propellant removal by machining. The first (September 1965 to February 1966) involved development of the TU-465.01, a 120 in. diameter segmented steel case motor. The propellant, catalyzed by ferrocene, posed special problems because of its impact sensitivity. During completion of propellant loading and curing operations, a combination of factors (contamination of liner surface, excessively hard propellant due to faulty formulation and processing) caused a massive separation around the aft port circumference and between the aft dome and grain. Using a modified Minuteman cutback machine fitted with special blades for cutting close to the motor wall, the propellant was removed to a depth of 26 in. from the face of the case bolt flange before the separation was completely removed and grain-wall bond integrity assured. After removing and recasting approximately 10,300 lb of propellant, this motor was successfully static fired.

Several years later (1967-1968) Thiokol built the TU-312L.02 demonstration motor, which had a 156 in. diameter segmented fiberglass case and a fixed ablative nozzle. Problems encountered with this motor included defective cast propellant in the forward motor segment.

To remove randomly oriented large voids, the segment was mounted vertically, and a Minuteman cutback machine was modified to perform the defect cutout. Blade configuration allowed an 80 in. diameter circular cut, but the machine was offset from the motor centerline so that an arc having 36 in. depth was cut into the web while the length of the cut into the segment was 133 in. (Figure 3-5). A total propellant weight of approximately 23,400 lb was removed. This machining was done dry and cutting blades making the outer periphery cut were curved to eliminate the stress rising effect of a sharp corner
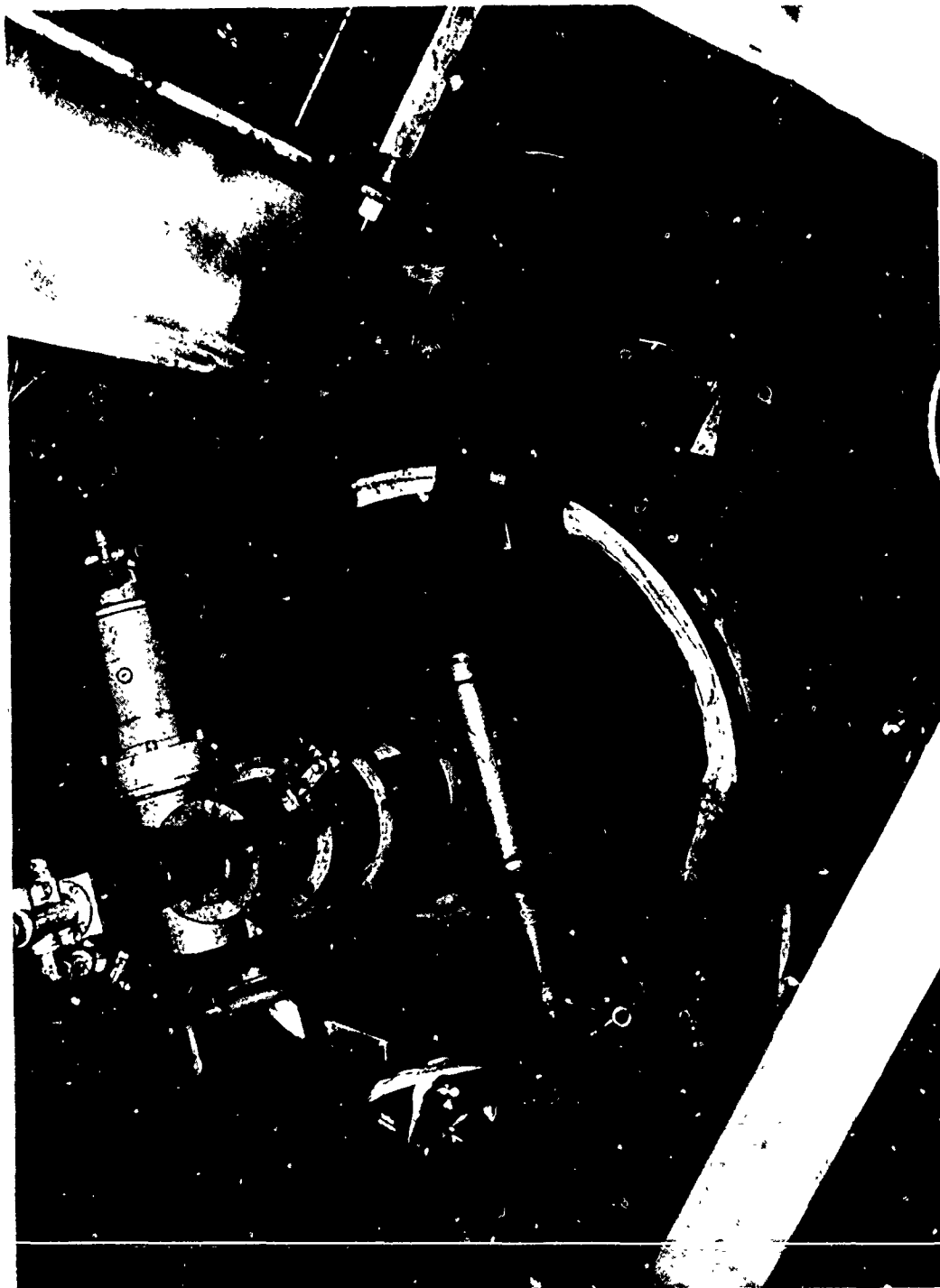
Figure 3-5. Minuteman Stage I Cutback Tool Modified to Remove Propellant From TU-312L.02

in the cutout cavity. All cutting operations were conducted remotely and monitored by television and audio systems.

The machining operation was finished by making skim cuts on all machined surfaces to remove contamination and to insure a good bonding surface for the recast propellant. The core was reinstalled and propellant was recast resulting in a successful static firing on 25 June 1968.

Thiokol was active in machining overcast propellant grains on the Minuteman Program. Cutback was required due to the nature of casting composite propellant. Even though Thiokol employs vacuum casting, small voids develop near the top of a propellant charge and tend to remain because there isn't enough static head pressure to compress them during cure. To correct this, Minuteman First Stage motors were overcast (casting vertical, aft end up) by an average of 400 lb, then the grains were machined primarily to obtain the desired grain shape but also to eliminate the propellant with voids. This operation was performed by Minuteman cutback machines which were designed specifically for this purpose, and by using remote monitoring and control. Thiokol's expertise in this operation is very extensive as 2,973 motors were processed. The cutback operations clearly indicated that dry cutting of Class 1.3 propellants is safe and technically acceptable as a method to remove propellant from rocket motors.

Cutback of the Thiokol Genie motor is performed primarily to obtain an exact grain length. The machined surface is coated with an adhesive liner material to prevent grain end burning. Then the motor aft plate/nozzle assembly is installed which mates with the liner.

Thiokol/Wasatch has extensive experience for machining propellant containing nitroglycerin, because Thiokol manufactures crosslinked double base propellant for the Trident-I (C-4) First Stage motor. The core is inserted into this motor

REFERENCES

1. C. D. Kalfadelis, "Development of Fluidized Bed Incinerator for Explosives and Propellants," Esso Research and Engineering Company, Trenton, New Jersey, October 1973.

2. V. T. Ciccone, A. P. Graves, J. S. Santos, R. Scola, "Economic Analysis of Rotary Kiln Versus Fluidized Bed P&E Incinerator," Technical Report AR LCD-TR-78033 ARRADCOM, Dover, New Jersey, September 1978.

3. R. A. Knudsen, "Hazards Analysis and Pollution Abatement Techniques," Contractor Report A0262-520-03-007, Picatinny Arsenal, Dover, New Jersey, June 1974.

4. G. Petimo, et al., "Detonation Propagation Tests on Aqueous Slurries of RDX, HMX, M-1 and Nitrocellulon," Contractor Report ARLCD-CR-77002, ARRADCOM, Dover, New Jersey, April 1977.

5. G. Petimo, et al., "Detonation Propagation Tests on Aqueous Slurries of TNT, Composition B, M-9 and M-10," TR-4584, Picatinny Arsenal, Dover, New Jersey, November 1973.

6. G. Petimo, et al., "Flow Characteristics of Explosive Slurries Injector System," Contract Report ARLCD-CR-77004, ARRADCOM, Dover, New Jersey, April 1977.

5.0 INSULATION REMOVAL AND REPLACEMENT TECHNIQUES

Salvaging of composite cases with insulation material having acceptable properties is possible by the removal of flaps and liner followed by thin replacement with new flaps and liner. This is a low risk, low cost procedure that was employed on Minuteman and C-4 Programs. Flap removal is accomplished with the use of heat and mechanical grinding techniques, while liner removal from the case insulation surface is a standard Thiokol rework procedure to repair liner application discrepancies. Solvents such as MEK, alcohol, and methyl-chloroform, plus hand scraping and mechanical abrading are all included in the process.

Salvaging flaps is impractical because their thinness leads to tearing and distortion. Also, in older motors the flaps are the most susceptible rubber components for age degradation.

Solvents and water present in internal insulation after removal of the propellant and liner can be removed by evaporation and drying. The original physical properties of 'the rubber will return when solvents are evaporated.

New flaps would be installed by the normal manufacturing process of secondary bonding with ambient temperature curing adhesive.

Installation drying procedures employed in present motor manufacture will be followed to remove any water or solvents absorbed into the insulation. This will prevent the inhibiting of liner and propellant cures.

Next in complexity of rework to the removal and replacement of flaps and liner only is the removal and replacement of this insulation between flap bulbs. This is believed to be feasible and of moderate risk. However, the insulation and insulation to case bond must be left intact between the bond area of the flap bulb and the case. Present work at Thiokol on a Thiokol AOTTS Third Stage Minuteman III and a 10-year-old

TABLE 3-3

SUMMARY OF LITERATURE SEARCH ON PROPELLANT CUTTING, REMOVAL, AND WASTE DISPOSAL

Literature Search Designation	Number of Initial Leads	Number of Apparently Relevant References	Number of Directly Applicable References*
A. Computer Based Searches Using Fixed Identifiers/Key Words			
Dialog File 6: NTIS 64-79/ISS24	903	102	19
NASA/RECON 32359	12	12	
NASA/RECON 44084	43	1€	5
NASA/RECON 44085	8	6	1
NASA/RECON 44097	23	13	6
Dialog CA Searches 77-80, 72-76, 67-71	13	8	4
Dialog NTIS 64-80/ISS17	42	23	3
DDC No. 089385	70	12	7
LMSC-NTIS 64-80/ISS03	23	7	1
(CPIA)CL1-TLR-450	5	5	5
B. Topical Survey of Chemical Abstracts and Current Journals	45	13	1

*Does not include several references on order and not yet received

2.   Using Cavitating Water Jets for Demilitariza-
tion, A. F. Conn and S. L. Rudy, Symposium on
Demilitarization of Conventional Explosives at
Naval Ammunition Depot, Hawthorne, Nevada, 20-22
Apr 1976.

Rocket Motor Case Reclamation, H. F. McQueen and J. C.
Ladd, Thiokol Corp., Wasatch Division, May 1964.
Comments: Historical. Describes early development of
hydromining techniques and parameters tested.

Final Report - Investigation of TU-465 Motor Propellant
Separation, TWR-1717 Project 3047, PMDI-66-7, D. C.
Bjorkman, E. D. Brown, D. W. Kase, Thiokol/Wasatch Divi-
sion, March 1966
Comments: Describes removal of 8-10,000 lb of propellant
from aft grain to eliminate a crack and repair motor.

Final Report, Task 8, Repair/Retrofit Procedures, SL/M
Program, VC 3T-T7-17-20, TD 8-79-7-8, TWR-2735, Thiokol/
Wasatch Division, July 1968.
Comments: Concepts investigated included:
1.   Propellant cutting to remove defects and/or
reduce stress
2.   Potting or inhibiting crack propagation
3.   Conditioning propellant surface with Freon
or other material
4.   Designing retrofit motor configuration
Cutting: By special milling, wire cutting, propellant
with rigid blade cutters are discussed.

Development of Cavitating Water Jet PCR Case Reclamation
Facility (U), Technical Report; IHSP-76-132, B. Skinner,
Jr., Hydronautics Inc., Laurel, MD, July 1976.
Comments: The Case Reclamation Facility reclaims rocket
motor casings by employing a high pressure water
jet system to erode away the propellant and

insulator contained within the casings. The
results of the initial phase of the contract,
which was to perform cutting tests on inert
samples of propellant and insulator with the
Cavijet at the contractor laboratory, were
promising. Unfortunately, the test results were
not very encouraging. Based on these results
and other problems associated with the employ-
ment of the Cavijet method at the case reclama-
tion facility, the rental or leasing of the
method would not be beneficial to the Navy.

Explosives Research and Development, October – December
1977 (U), No author cited, Naval Weapons Center, China
Lake, CA, December 1977, Confidential Document.

Investigation of Underwater Burnout as a Means of Reclaim-
ing Metal Parts From Rejected Pershing Motors, Thiokol
Corporation/Alpha Division, Huntsville, AL, U-A-62-272A.
Comments: A study was conducted to evaluate the feasibil-
ity of reclaiming the metal parts of rejected
Pershing motors by burning out the propellant
while the motor was submerged in water. The
metal parts of a Pershing size motor could be
reclaimed by underwater burnout, depending on
the severity of the defect. A description of
the underwater burnout facility is included.

Hydraulic Removal of Propellant From Rocket Motors for Case
Reclamation, M. H. Larimer, Thiokol Corporation, Redstone
Division, Huntsville, AL, Report Number U-A-62-145A,
30 Apr 1962.
Comments: Hydraulic removal techniques have been developed
for the cleaning of casting cans to replace the
cleaning-by-hand methods previously used.

Degraining--A Three-Step Process to Obtain Propellant
Samples From Case-Bonded Motors (U), L. G. Pridy, J. W.
Sebert, Naval Ordnance Station Indian Head, MD, Report
IHTR-417, March 1975.

Comments: A three-step degraining process has been devel-
oped to obtain propellant samples from case
bonded motors for chemical/physical tests. The
three steps are electrolytic machining, section
removal by piano wire cutting, and propellant
removal by piano wire cutting.

Final Report - SRAM Case Reclamation, M. J. McIntosh,
Thiokol Corporation/Wasatch Division, Report TWR-1346, 24
Feb 1972

Comments: Applicable. Portable washout equipment used.

Abstract: SRAM cases were reclaimed by subcontractor
(Byron Jackson Company) using a portable washout
system under Thiokol direction. Ignition oc-
curred during the first washout. The remaining
four cases were successfully reclaimed.

Final Report - Poseidon Case Reclamation, M. J. McIntosh,
Mfg Engr Report 1329, Thiokol Corporation/Wasatch
Division, 20 May 1971.

Comments: Applicable. Composite glass case reclaimed

Abstract: Propellant was removed from a first stage Posei-
don case. Slight damage of insulation was re-
parable and the case was reused for casting.
Steam was used to soften the propellant allowing
use of low pressure (3,000 psi) water for
cutting.

Final Report - Investigation of High Pressure Water Noz-
zles, M. J. McIntosh, Thiokol Corporation/Wasatch Divi-
sion, Mfg Engr Report 1096, 1097, July 1965.

Comments: Applicable. Basic nozzle technology

Abstract: Investigation of nozzle efficiency, stream sta-
bility and water velocity of various types of
nozzles. Factors affecting cutting capability
of the water jet are listed.

Final Report - Solid Propellant Waste Disposal/Ingredient
Recovery Study, M. J. McIntosh, JPL Contract 954161A,
Thiokol Corporation/Wasatch Division, 4 May 1976.

Comments: Applicable. Waste Disposal

Abstract: Study conducted to define economic and energy
related aspects of waste rocket propellant dis-
posal. Comparisons of facility and operating
costs shows open burning to be lowest cost meth-
od of incineration. Recovery of ingredients in
larger program has possibility of being profit-
able.

Minuteman II Stage III Propellant Removal, Ogden ALC/Aero-
jet General Corporation, Contract F42600-79-C5618, 26 Nov
1979.

Comments: Applicable

Abstract: Final Report not completed.

3.4 CONCLUSIONS

The history of rocket motor propellant mechanical cutting
methods clearly indicates that the method is proven technology
which should be considered as a method of propellant removal
when reclaiming rocket motor cases. The capability of locating
the cutting surface precisely makes it desirable when we are
trying to prevent damage to the insulation or the case.

4.0 WASTE PROPELLANT DISPOSAL TECHNIQUES

Waste propellant and waste explosive disposal methods were assessed by reviewing available literature and by contacting several industrial and military installations. Contacts were made with ARRADCOM at Dover, Joe Santos; Radford Arsenal in Virginia, John Horvath; Army Arsenal Illinois, Bob Lindholm; Tooele Army Depot, Frank Crist. Also, Mr. John Brown of John Brown Associates, Inc. was contacted. Mr. John Brown was under contract by ARRADCOM to study the alternatives to incineration of bulk explosives, propellants and pyrotechnics DAAK11-78-C0123. He has submitted his report dated October 1979.

There are a number of ideas and some experimental work being accomplished at the present time. Three pilot plants are currently evaluating: (1) rotary kiln incineration, (2) wet air oxidation, and (3) fluidized bed incineration.* Fluidized bed incineration is being further evaluated by the Tooele Army Depot contracts (who received proposals from contractors on 8 Dec 1980).

Several unique methods of waste propellant disposal are considered. One is to sell the waste to an acceptable buyer who in turn would use the material to make industrial products such as mining explosives or primary raw materials. Also, studies have been conducted and patents issued regarding conversion of the hazardous waste to primary raw materials such as NH_4ClO_4 and aluminum.**

Thiokol is working in conjunction with a blasting supplier that supplies blasting agents to the mining industry (Kennecott). Radford is working with suppliers that supply blasting agents to the coal mining industry.

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*Santos, Joseph S. and John J. Conavan: "Incineration Processes for Propellant and Explosive Waste Disposal," Facilities and Protective Technology Division, Manufacturing Technology Directorate, Picatinny Arsenal.

**McIntosh, Meldon J., Solid Rocket Propellant Waste Disposal/ Ingredient Recovery Study, Final Report JPL Contract 954161A, 4 May 1976, Thiokol Corporation, Wasatch Division, Brigham City, Utah.

To date, the method used by most rocket motor manufacturing industries is open air burning. The cost of open air burning varies from $0.05 per lb to about $2.00 per pound. The higher costs are attributed to propellant packaging and transportation to remote areas where burning is allowed. A propellant producer in California pays the premium because of the EPA restrictions in their area.* The referenced document defines the energy and cost related aspects of waste rocket motor propellant disposal.

4.1 APPROACHES USED BY VARIOUS ARMY AGENCIES AND ARSENALS

The week ending 23 November and 30 November, several telephone calls were made to various propellant manufacturing companies and U.S. Arsenals. The objective of these telephone calls was to locate and visit areas with unique disposal systems.

4.1.1 ARRADCOM

Mr. J. Santos indicated that the ARRADCOM does not specifically have an operational disposal system at the present time. They do have a unit that is approximately 5 ft in diameter with a bed depth of about 8 ft in which data have been accrued. Mr. Santos indicated that the major problem with the fluidized bed incinerator was the feeding problems. He recommended publications:

1. Fluidized Bed Incinerator for Disposal of Propellants and Explosives, Technical Report ARLCD-TR-78032, October 1978.
2. Evaluation of Incinerator for Waste Propellants and Explosives, Technical Report 4984, Picatinny Arsenal, Dover, NJ, December 1976, DTICAL.

The above incinerator reduced approximately 200 lb of propellants and explosives per hour. Mr. Santos indicated that

*McIntosh (as cited on page A-42)

the Army had a large contract to examine incinerators that many independent firms would be bidding on. Mr. Santos also mentioned the difference between the rotary kiln disposal methods at Radford and Tooele is that the Tooele plant rotary kiln was fabricated from 3 in. armor plate and was a popping furnace. The Radford unit was lined with firebrick and only accepted explosive stock that was not contaminated with metals.

At the present time all of the Dover waste materials, propellants and explosives, are being destroyed by open air burning.

4.1.2 Illinois Army Arsenal

Mr. R. Lindholm, maintenance operations, is in charge of the destruction of explosives at the Illinois Arsenal. He has been working with Mr. Santos of ARRADCOM on fluidized bed systems. His plant, in conjunction with the Tooele, Utah Arsenal, originated the fluidized bed evaluation contract. Mr. Lindholm had trouble earlier in the year with fluidized bed concept, since his feed stock needed to be reduced in particle size to less than 10 mesh to obtain a slurry, he was having trouble grinding the material. Thiokol recommended using high pressure water cutting nozzles to reduce the material.

Mr. Lindholm indicated that Aerojet people were going to use their experience in reducing nuclear waste to springboard them into a fluidized bed incinerator for propellants and explosive waste. Tom Harrington of the Aerojet Sacramento Propellant Plant was in charge of the engineering development work of their pilot plant unit. Mr. Lindholm said there was a bibliography from the Cameron Station on explosives incineration and grinding, which had all the published information from 1960 to date on these two subjects.

4.1.3 Aerojet Propulsion

Mr. J. White of Aerojet indicated that Thiokol could not view Aerojet's fluidized bed and incinerator system; however, a visit may be arranged for Aerojet's nuclear waste fluidized bed

incinerator facilities and nuclear waste reduction plantsite.
Their pilot studies on the fluidized bed incinerator consisted
of a 12 in. diameter bed, approximately 6 ft deep. Its perform-
ance, consumption rate, etc. are not yet known.

4.1.4 Aerojet Energy Conversion Company

Mr. Frank Ulbrich works for the Aerojet Energy Conversion
Company whose major business is fluidized bed dryer incinerator
volume reduction systems. Aerojet proposed to the Tooele Ord-
nance Depot a unit with a small 12 in. diameter fluidized bed
that is being set up at the Aerojet Rocket and Propellant plant
at Sacramento. This unit would be used in Phase I of their
proposed program which would consist of trial runs of explo-
sives into the incinerator to determine the feed rate and part-
icle size of the explosive necessary to obtain uniform combus-
tion. The water to explosive ratio or water to fuel ratio
necessary to sustain a uniform combustion and uniform gas flow
through the fluidized bed and other tests would be conducted to
determine optimum bed temperatures for explosive incineration.
With these data a pilot-sized fluidized bed system would be set
up for Phase II. The pilot unit would be used to gain data on
equipment size, equipment support requirements, ash removal of
gas controlling systems such as scrubbers, precipitators, etc.
The question regarding oxides of nitrogen as produced by the
incinerator was answered "the use of nickel catalysts in the
fluidized bed will reduce, if not eliminate, the formation of
the nitrogen oxide systems (NO_x)." Mr. Ulbrich was quite
sure that the use of nickel catalyst in the combustion sequence
in the fluidized bed was sufficiently proven that it was a
state-of-the-art method of controlling NO_x emissions. The
pilot plant data would then be gathered on sustained burning of
propellants to determine optimized bed versus pound per hour
incineration rates of explosives.

The pilot plant would also be used to determine the sup-
port equipment--scrubbers blowers, ash removal--etc. for

the system. Mr. Ulbrich indicated that a fluidized bed incineration system would safely incinerate production scrap propellant and other explosives at a cost of around $0.40 to $0.50/lb. He indicated that the rotary kiln system presently used at the Tooele Ordnance Depot and at the Radford Laboratories would cost in the range of $0.75 to $1.00/lb for waste propellant and explosive incineration. He indicated that at the present time, the method of incinerating and/or discarding explosive or flammable waste from Aerojet Propulsion costs about $2.00/lb for shipping the material into a county where it can be burned. Aerojet is forbidden by the EPA to open air burn their propellant and explosive wastes in the county where their plant is located.

4.1.5 Radford Army and Munition Plant

Mr. John Horvath at the Hercules GOCO Plant at Radford Army and Munition Plant in Radford, Virginia, indicated that he does have a refractory lined rotary kiln incinerator that is operational at Radford. The unit is used to burn propellants and explosives when they contain no metal. This is obviously a requirement to keep high velocity metal particles from breaking up the refractory lining. He indicated that they do incinerate many of the propellants and explosives in the unit but it handles no more than 5% of the propellant and explosive waste at Radford. They are still conducting feed, admission, and efficiency studies on the operation of the equipment. They are currently working with blast supplies around Virginia to put their propellant and explosive waste in a slurry form to be used in the local mining industry.

4.2 LITERATURE SEARCH

1. Sensitivity and Characterization of Liquid Ammonia Systems: Reclamation Methodology for AP Propellants - IT-M57-17-42 (Liquid Ammonia and Solvent Dissolve AP) Reference TC Work

2. Dissolution of Solid Propellants or
   Polymers - IT-046-4-653 OPI (NASA)
3. Wet Oxidation Incineration - Indian Head,
   Maryland - IT-M54-26-1 "Propellant
   Disposal/Reclamation Faulty Design," 1974
4. Environmental Impact for Disposal of
   Propellant and Ingredients TI-0581-48-74-2
5. Waste Water Treatment EPA - Explosive and
   propellant Volume III - IT-0808-53-67
6. Recovery of NG - IT-037-4-203 Literature
   Search by J. C. Hindshaw (LMSC) 1979
7. 1975 Literature Search Reclamation of Waste
   Propellants, NASA - IT-046-4-415
8. Lab Study of Pyrolysis of Explosives
   Contaminated Georgia Institute of
   Technology - IT-0159-17-11
9. Microfilter - AD-A027 329 - Rensselaer
   Polytech Institute - Treatment of Waste
   Water - EXP and Propellants, Troy, New
   York
10. Microfilter - N79-10227 Leaching AP From
    Propellant - Graham Shaw

4.3 ABSTRACTS

IT-M94-17-1   AD A042601

"Toxicological Investigation of Pilot Treatment
Plant Wastewaters at Holston Army Ammunition Plant,"
G. M. Stilwell, et al., Battelle, Columbus
Laboratories, Columbus, Ohio, July 1977.

Describes bioassay tests conducted on HAAP waste
water. Overall results indicated that biological
treatment, either activated sludge or the com-
bination trickling-filter-activated sludge does
reduce the toxicity of manufacturing waste water.

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If chemical dissolution of HMX from HE propellant were used, reclamation of HMX could produce contaminated waste water. This provides a method of treatment of the contaminated water.

IT-0159-17-11   AD A058006

"Laboratory Study of Pyrolysis of Explosive Contaminated Waste," J. A. Knight and L. W. Elston, Georgia Institute of Technology, Atlanta, Georgia, July 1978.

Pyrolysis of mixed waste containing 2% TNT produced storeable and transportable fuels, char and oils, recovering about 70% of the energy input. Gases also produced which account for 16-22% of the energy input. No explosion hazard evidenced at 650°C decomposition temperature.

Possibly applicable to disposal of waste during propellant removal.

IT-0808-33-23   PB 258518

"Report to Congress on Hazardous Waste Disposal," Environmental Protection Agency, Washington, D.C., January 1973.

Not directly applicable. Generally concluded that management of all wastes were inadequate and that the magnitude of the problems was increasing.

Cites cost of treatment/disposal processes (1973 dollars):
$1.40/ton for carbon sorption
$10/ton for neutralization/precipitation
$13.60/ton for chemical oxidation
$95/ton for incineration

Gives flow diagrams and cost estimates for several
waste disposal concepts of 76 references cited, the
most applicable were:

1. Swift, W. H., "Feasibility Study for Development
   of a System of ...,"
   U.S. Environmental Protection Agency Contract
   68-06-0762, Battelle Memorial Institute, 1 March
   1973.

2. Ohinger, R.S., "Recommended Methods of
   Reduction, Neutralization; Recovery or Disposal
   of Hazardous Wastes," Volume 1, USEPA Contract
   68-03-0089, TRW Systems Group, Inc., June 1973.

3. Booz, A., "A Study of Hazardous Waste Materials,
   Hazardous Effects and Disposal Methods," USEPA
   Contract 68-03-0032, Applied Research Institute,
   June 1972.

IT-M17-17-4   NSWC/WOL TR 77-72

"Utilization and Disposal of Solid Propellant
and Explosive Wastes," A. S. Tampa and D. M. French,
Naval Surface Weapons Center, White Oak Lake,
Maryland, April 1977.

Very applicable describes simple methods for
breakdown of crosslinked composite solid propellants
and explosives and recovering their constituents for
use.

IT-M17-17-5   NSWC/WOL TR 77-72, Appendix A, December 1977

Addendum to above contains detailed calculations
of costs. Thiokol's water extraction process is
cited.

IT M54-26-1-2   AD 916 820L

"Industrial Preparedness Measure: Propellant
Disposal/Reclamation Facility Design"
IHMR 73-240, K. L. Wagaman and T. J. Sullivan Naval

Ordnance Systems Command, Indian Head, Maryland,
September 1973

Study made to determine the maximum water
content of waste propellant slurries that can be
used in incineration units and wet oxidation
reactor.

IT 0231-29-2   PB 256921

EPA Contract 68-03-0089
"Hazardous Waste Disposal Program," 6th Monthly
Report, July 1972.

Contains process report on pyrolysis and
references which may be useful.

AD-A064124   Army Armament Research and Development Command

"Fluidized Bed Incineration for Disposal of
Propellants and Explosives, Etc. (U)," October 1978,
R. Scola, J. S. Santos. Fluidized bed chosen as
best method of incineration for propellant and
explosives. Detonation propagation tests were
conducted.

Fluidized bed incineration chosen due to its
reported characteristics of high combustion
efficiency, low emission, high heat sink capacity,
low operating cost and inherent safety features.

Successful completion of tests at the 22 wt
percent slurry concentration level displayed
capability of fluidized bed incinerator to comply
with 200 ppm goal of NO_x and other gaseous
emissions.

MHSMP-76-51

"Disposal of Waste or Excess High Explosives,"
Final Report Mason and Hanger - Silas Mason Company,
Inc., Amarillo, Texas (U), January 1977.

Tested Rotary Kiln Incineration: discusses
flash-back versus feed rate tests. Also discusses
closed pit batch-type incineration.

Both concepts are feasible but a greater effort
would be required to develop rotary kiln method.

Report lists advantages and disadvantages of
open burning, detonation, incineration, deep well
injection, ocean dumping, biochemical decomposition,
and chemical recovery. Chooses incineration as best
method. Not very specific about the "how" of each
method. Gives results of incineration test but no
cost data.

PB-296 642

NSF/RA-790046 "Immobilization of Hazardous
Residual by Encapsulation," R. V. Subramanian, et
al., Washington State University, Pullman,
Washington, February 1979.

Demonstrates feasibility of encapsulation of
hazardous wastes (particularly radioactive) in
aqueous slurries in water-extensible polyester
matrix. Two and one-half times more expensive than
cement silicate encapsulation.

PB-279 773   EPA/530/SW-157C

"Economic Impact Analysis of Anticipated
Hazardous Waste Management Regulation of the ...,"

Daniel W. Franke, et al., Development Planning
and Research Association, Manhattan, Kansas,
February 1978.

Not applicable. Relates to leather industry wastes.

PB-279 645   EPA/530/SW-158C

"Economic Impact Analysis of Anticipated
Hazardous Waste Regulation on the Industrial ...,"
J. Stollman, et al., Energy Resources Company, Inc.,
Cambridge, Massachusetts, January 1978.

Only slightly applicable. Includes explosives
manufacturing as part of organic chemical industry.
Projects effect of disposal on overall costs.

PB-265 042   EPA/600/2-76/213C

"State-of-the-Art: Military Explosives and
Propellant Production Industry (Volumes I, II and
III)," James Patterson, et al., American Defense
Preparedness Association, Washington, DC, October
1976.

Study surveys military explosives and propellant
manufacturing industry, covering both "GOGO" and
"GOCO" facilities. Sources of waste water, volumes,
and pollutant constituents have been reported.

Treatment technology currently in use at various
installations have been examined and evaluated. The
report consists of these volumes:
Volume I - General conclusions and recommendations
and describes manufacturing operations.
Volume II - Bulk of data concerning waste water and
treatment systems.
Volume III - Reviews and summarizes data from above
and evaluates new treatment processes under
development.

PB 246727

"Chemical Waste Incinerator Ship Project,"
Volume I, 231 pp, Maritime Administration, Washington, DC, Environmental Activities Group, MA
EIS 7302 76 08DI, 1975.

PB 246 728

Volume II, 221 pp.

PB 253 978   MA-EIS-7302-76041-F

Volume 1, 1976

PB 253 979   EPA/430/9-75/014

Volume 2, 1975

Not especially applicable except as an alternate method of disposal.

Related to the growth of the chemical industry has been the accumulation of the ever increasing volume of toxic chemical wastes such as chlorinated hydrocarbons. A relatively environmentally safe disposal method for toxic chemical wastes, which are liquid and combustible, is incineration at sea.

AD-A024 513   Picatinny Arsenal, Dover, New Jersey

"Development Trends in the Incineration of Waste Explosives and ...," (U)   I. Forsten, J. S. Santos, R. Scola, May 1976.

A review of development in explosive and propellant waste incineration processes is presented which includes a vertical induced draft system, rotary kiln, simplified incineration techniques for pollution abatement I and II, wet air oxidation, and fluidized bed incineration.

Advantages and disadvantages of each concept are discussed including efficiency, relative costs,

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environmental effects, flexibility of operation and
safety aspects. Design background and status of
pilot plant development of the fluidized bed
system is included.

PB-261 086   EPA/530/SW-171

"A Summary of Hazardous Substance Classification
Systems," Allen M. Kohan, Environmental Protection
Agency, Washington, DC, 1975.

Slightly significant for information.

This paper describes the criteria used by 23
systems to define a "hazardous substance," primarily
for regulatory purposes.

4.4   CONCLUSIONS AND RECOMMENDATIONS

Disposal state-of-the-art technology is limited to open
air burning. Minor data are available for pilot plants on
rotary kiln disposal operations and fluidized bed incinerators.
Other methods or ideas are experimental stage only. Cost
evaluations for any method other than open air burning at the
present time are limited to knowledgeable people's evaluation.

It is recommended that studies above and beyond the scope
of this program be initiated to provide feasible alternatives
to the disposal of waste propellant and explosive materials.

REFERENCES

1. C. D. Kalfadelis, "Development of Fluidized Bed Incinerator for Explosives and Propellants," Esso Research and Engineering Company, Trenton, New Jersey, October 1973.

2. V. T. Ciccone, A. P. Graves, J. S. Santos, R. Scola, "Economic Analysis of Rotary Kiln Versus Fluidized Bed P&E Incinerator," Technical Report AR LCD-TR-78033 ARRADCOM, Dover, New Jersey, September 1978.

3. R. A. Knudsen, "Hazards Analysis and Pollution Abatement Techniques," Contractor Report A0262-520-03-007, Picatinny Arsenal, Dover, New Jersey, June 1974.

4. G. Petimo, et al., "Detonation Propagation Tests on Aqueous Slurries of RDX, HMX, M-1 and Nitrocellulon," Contractor Report ARLCD-CR-77002, ARRADCOM, Dover, New Jersey, April 1977.

5. G. Petimo, et al., "Detonation Propagation Tests on Aqueous Slurries of TNT, Composition B, M-9 and M-10," TR-4584, Picatinny Arsenal, Dover, New Jersey, November 1973.

6. G. Petimo, et al., "Flow Characteristics of Explosive Slurries Injector System," Contract Report ARLCD-CR-77004, ARRADCOM, Dover, New Jersey, April 1977.

5.0 INSULATION REMOVAL AND REPLACEMENT TECHNIQUES

Salvaging of composite cases with insulation material having acceptable properties is possible by the removal of flaps and liner followed by thin replacement with new flaps and liner. This is a low risk, low cost procedure that was employed on Minuteman and C-4 Programs. Flap removal is accomplished with the use of heat and mechanical grinding techniques, while liner removal from the case insulation surface is a standard Thiokol rework procedure to repair liner application discrepancies. Solvents such as MEK, alcohol, and methylchloroform, plus hand scraping and mechanical abrading are all included in the process.

Salvaging flaps is impractical because their thinness leads to tearing and distortion. Also, in older motors the flaps are the most susceptible rubber components for age degradation.

Solvents and water present in internal insulation after removal of the propellant and liner can be removed by evaporation and drying. The original physical properties of 'the rubber will return when solvents are evaporated.

New flaps would be installed by the normal manufacturing process of secondary bonding with ambient temperature curing adhesive.

Installation drying procedures employed in present motor manufacture will be followed to remove any water or solvents absorbed into the insulation. This will prevent the inhibiting of liner and propellant cures.

Next in complexity of rework to the removal and replacement of flaps and liner only is the removal and replacement of this insulation between flap bulbs. This is believed to be feasible and of moderate risk. However, the insulation and insulation to case bond must be left intact between the bond area of the flap bulb and the case. Present work at Thiokol on a Thiokol AOTTS Third Stage Minuteman III and a 10-year-old

Aerojet surveillance motor AGC-30018 has been conducted to remove NBR insulation in the cylinder area of the case. This was accomplished by peeling strips of insulation while locally heating the V-57 tie cement between the insulation and fiberglass case with a hot air gun. The NBR insulation cleanly separated from the case but the V-57 Ty cement remained to contaminate the fiberglass bonding surface. Recently it has been demonstrated on a postfired Third Stage Minuteman III that this V-57 Ty cement can be removed with a 130°F 3,000 psi hydrowashing process, further work with this process is necessary to determine the degree of risk. Experience for this type of rework also includes the removal and replacement of a hydrotest rubber bladder and the addition of cured segmented insulation in a 156 in. TU-312 motor* for static test. Similar removal and replacement of poorly bonded cylinder insulation was accomplished in an MX Kevlar composite case. In the MX motor, longitudinal strips of cylinder insulation were bonded with UF-3195 at 135°F for 6 hr. This was a low risk inert motor.

Although solvents are used to clean subsequent bonding surfaces and remove rubber to salvage metal parts, our technical assessment is that exposure of the composite case to solvents should be held to a minimum because of the porous nature of the composite, wicking by the filament, and degradation of the resin matrix. It is not recommended to employ solvent soaking to remove insulation material.

A second step in the complexity of removal of insulation and rework is the removal and scrapping of liner, flaps, and thin cylinder section insulation followed by the mechanical removal of unsatisfactory surface thickness of dome insulation. This mechanical insulation removal in dome areas would be restricted to areas away from metal polar bosses, that is, nozzle-insulation interface and igniter-insulation interfaces.

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*Demonstration of 156 in. motor with segmented fiberglass case and ablative nozzle, AFRPL-TR-68-159, Vol I, 1968

Also mechanical insulation removal in dome areas would be restricted to leave a minimum of 0.060 in. of original insulator thickness bonded to the case both in flap bulb bond area and areas exposed any significant time during motor firing. The risk to motor operation and safety factors is believed to be too great for complete insulator removal in dome areas because of necessary joints in insulation rework and likely void sizes in secondary bonding operations.

The replacement of flaps and liner only can be accomplished by methods followed in the original manufacture of Third Stage Minuteman III, C-4, and MX motor cases (Fiberglass and Kevlar Composite). Steps include: (1) dryfitting flap, (2) abrading, solvent cleaning, and drying flap and case insulation, (3) bonding flap to case insulation with ambient or low temperature curing epoxy adhesives, (4) recleaning installed flap and case insulation, and (5) applying and curing liner. This replacement would be low risk and low cost as this is already a standard procedure.

The second mode of rework would be the above work preceded by the replacement of thin insulation in the cylinder section of the motor. This thin insulation will be pre-cured and then secondarily bonded in place with adhesives requiring ambient curing or cure temperatures that will not degrade the composite case. The work has been done on the previously mentioned 156 in. TU-312 motor and MX motor cases.

A third more drastic mode of rework of insulation to salvage composite cases is the removal of liner, flaps, and thin insulation in the cylindrical case section, followed by grinding to remove age affected dome insulation. Grinding of dome insulation must be restricted to not alter insulation at the polar bosses, igniter-insulation interface, nozzle-insulation interface, or insulation within 0.060 in. of composite case inside mold line. Segmented dome insulation additions to obtain required insulation thickness would be pre-cured to the proper geometry. These would then be secondarily bonded

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with adhesives. The choice of adhesives would be limited
to those that cure at ambient temperature or elevated
temperatures compatible with composite case materials.
Sectioned thin insulation in the cylinder area would be
installed in a similar manner. The flaps and liner should be
installed in a manner consistent with original manufacturing
procedures.

5.1 LITERATURE SEARCH

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0317-22-1045, 7pp, 6 February 1968

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L. Evans, TC (Wasatch), Final Report, "Evaluation of Repair of
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11623, AD 602 632, (80pp), 20 June 1964

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"Water Jet Cutting Technical and Economic Feasibility
Study," D. B. Hibshman, Thiokol Corporation/Wasatch
Division, March 1980
Program plan initiated to evaluate cutting of uncured
rubber and uncured composite materials by high velocity
water jet. Technology may be useful in general area of
case salvage.

AFRPL-TR-68-159 Volume I TCO-56-9-8

"Final Report - Detonation of 156-In. Motor," the 156
in. demonstration motor incurred many problems applic-
able to salvaging glass/Kevlar motor cases and to
reinstallation and/or repair of liner and insulation.

1T-01-1-5-2   AD 442761

"Polaris Power Plant Development Addendum,"
Report 3688-31M-5, March 1964, Aerojet General
Corporation.

Contains information on development of Rapid-
Curing Bonding Systems for internal insulation.
Some of this may be applicable to insulation removal
and reinstallation of the insulation in the case.

1T-T7-15-154   Manufacturing Engineering Report 1413

"C-4 Insulation Cold Patch Repair Study," V. C. Goodey, Thiokol Corporation/Wasatch Division, September 1979.

Process developed for making acceptable cold patch repair on C-4 insulators. May be applicable to reinstallation of salvaged cases.

1T-T7-17-563   Manufacturing Engineering Report 1310

"Third Stage V-45 Rubber Repair Study," D. C. Merrill, Thiokol Corporation/Wasatch Division, August 1970.

Study conducted to determine what effect repair of a defective area would have on the physical properties of V-45 rubber in a Third Stage Minuteman motor. Results inconclusive. May provide insight into insulation removal and reinsulation of salvaged cases.

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"Stage III Minuteman Boot Nipple Repair Procedure," L. E. Jensen, June 1979.

May obtain information useful to reinsulation.

1T-T7-17-486   Manufacturing Report 1269

"Study to Define the Cause of Soft Spots in UF-1120 Insulation."

Possibly applicable to insulation removal and reinsulation.

6.0 CASE STRUCTURAL CONSIDERATIONS

To meet criteria in this area, the case reclamation process cannot reduce the case strength below the original design requirements. Experience with the reuse of composite cases is limited, but the results from the successful tests of a 156 in. fiberglass case and a 30 in. Kevlar case indicate the feasibility of case reclamation. In 1968, a Thiokol 156 in. diameter fiberglass (S-994 HTS glass roving with epoxy resin) segmented case (TU-312) was successfully hydroproofed three times, static fired, and finally hydroburst.

The first hydroproof was conducted to verify the design and fabrication. The second was to verify a rework to the skirt structure and a third to verify a rework to the rubber insulation. After static test the insulation was cleaned up, new rubber insulation added, and the case was finally successfully burst tested at a pressure of 1.29 x MEOP.

No equivalent experience has been obtained for cases made of Kevlar. However, a 30 in. diameter Thiokol Antares III motor case (Kevlar-49 fiber, epoxy resin) was proofed twice and structurally tested before it was finally hydroburst successfully. This experience coupled with the fact that C-4 motor cases (Kevlar-49 fibers) are now allowed two proof cycles prior to delivery would support the feasibility of multiproof testing of Kevlar cases.

One of the major criteria for selection of a case salvaging method is its effect on the structural integrity of the case. The following items must be considered before methods can be selected: (1) effects of broken fibers; (2) fiber matrix contamination; (3) multiproof testing, and (4) distortion of case geometry. The unique characteristics of a filament wound case are attributed to both the design approach and method of fabrication.

6.1 CASE DESIGN

There are basically two types of composite case designs used in industry today: (1) polar and (2) helical. The first type uses a polar or planar wound pattern for longitudinal strength with hoops overwrapped or interspersed for circumferential support. This consists of winding the rovings (groupings of fibers) on the mandrel in great circles, or more particularly, the filament path over the mandrel will be a straight line when viewed from the side.

The second type of design uses a helical wound pattern for longitudinal strength and hoop windings for circumferential support. These two patterns, as was the case for the polar design, may be segregated and/or interspersed. The helical pattern involves winding the rovings on the cylindrical section of the mandrel such that a curve is traced on the cylinder by the rotation of a point crossing its right sections at a constant oblique angle. The pattern in the dome sections of this design is usually geodesic in nature. This type of design is commonly used when the polar openings are large and when the L/D (case boss to boss length over case diameter) is large, such as the First and Third Stage Trident-I (C-4).

The fiber stress is calculated, neglecting the effects of the resin and, therefore, only undamaged fibers are considered.

The helical/polar fiber stress (σ_{α_f}) and hoop fiber stress (σ_{θ_f}) can be obtained from the following equations:

$$\sigma_{\alpha_f} = \frac{p\ R\ (1 + \varepsilon_\theta)}{2\ t_{\alpha_f}\ \cos^2 \alpha}$$

$$\sigma_{\theta_f} = \frac{p\ R\ (1 + \varepsilon_\theta)\ (1 - \frac{1}{2} \tan^2 \alpha)}{t_{\theta_f}}$$

P   = Case pressure

R   = Average case radius

ε_θ = Hoop strain

α = Helical polar wind angle

$t_{\alpha f}$ = Thickness of the undamaged helical/ polar fibers

$t_{\theta f}$ = Thickness of the undamaged hoop fibers

The remaining margin of safety (MS) of the case pressure vessel versus structure can be calculated using the following equation:

$$MS = \frac{\text{Allowable fiber stress}}{(\text{Actual fiber stress})\ (\text{required factor of safety})} - 1$$

The reduction in the margin of safety due to hoop or helical fiber damage can be directly determined if the extent of damage (number of filaments, layers) is quantitatively unknown.

To more accurately determine the effects of broken fibers on case integrity, the stress field in the area of the damaged fibers has to be known. In the "Y" joint area (where the skirt interfaces with the case), for example, consideration should be given to the bending discontinuities that may be present due to geometric nonlinearities and moment loads resulting from nonlinear load paths. The resulting stresses from this condition increase as a function of the thickness squared as compared with the linear relationship in the other areas as predicted by netting analysis.

Inasmuch as the cases involved in the reclamation program will incur much more handling and processing than was initially envisioned during design, consideration should be given to protect the cases during the time it will be out of service. The concern here is that composite cases are in general more susceptible to impact (handling or processing) damage than metal cases. The damage could occur in the form of resin-fiber shattering, and may not be readily noticeable during a visual inspection. Kevlar and rigid resins are more susceptible than

glass and flexible resins. Kevlar fibers are also very suscep-
tible to strength degradation as a result of its highly ori-
ented structure and the fact that the outer portion of the
fiber is more oriented than the inner portion. In addition, to
this condition, Kevlar is very susceptible to abrasion and
fraying during machining and handling because of its
subfilament microfibrillar structure.

The damage in the form of resin-fiber shattering, being
generally very localized, should not significantly affect the
margin of safety of the case (i.e., the load is "netted" around
the damaged area). The other damage conditions should be fur-
ther assessed in light of its effect on the strength of the
case and its effect on the subsequent reclamation procedures
(i.e., bonding of the insulation to the case may be hampered by
the fraying of the Kevlar).

6.2 CASE FABRICATION

When the case is wound, the impregnated fibers are preten-
sioned to provide a snug fit between the insulated mandrel and
the winding material. This creates a good bond when cured but
creates problems when removing the insulator in a case recla-
mation effort. This degree of bond, coupled with the following
facts will be considered to define technical requirements for
an acceptable case salvage method.

The resin content of the composite is kept as low as pos-
sible to increase the effective strength of the fibers. This
condition decreases the transverse properties of the com-
posite.

The effects of the low resin content on the transverse
properties are further increased in cases made from Kevlar-49
Aramid fibers. The chemical structure of this nonisotropic
fiber indicates the transverse properties are initially weak
because of the weak hydrogen bonds between polymer chains.
Cases made of glass do not have this condition because of the
high crosslinking of the isotropic glass and the stronger bond
it has with the resin system.

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The lower shear strength of Kevlar relative to glass makes it more sensitive to damage when the insulator is removed.

Fibers

Most of the operational and new composite case designs employ either "S" type fiberglass or Kevlar-49 fibers. The chemical structure and the sensitivity of each to processing and ambient environments are different and must be considered in processing a composite case. These properties and characteristics are compared in Table 6-1 and it is obvious that Kevlar is the most sensitive due to its poor abrasion qualities, water absorption characteristics, and low resistance to acids and bases.

TABLE 6-1

FIBER COMPARISON

	Kevlar-49	"S" Glass
Structure:	Anisotropic (Aramid)	Isotropic (glass)
Strength:	Good tensile up to 350°F	Good tensile beyond 350°F
	Low shear and compression	Good shear and compression
	Low abrasion	Good abrasion
Chemical:	7% water absorption	Inert
	Affected by acids and bases	

Recently, it has been identified that the use of the silicone release agent (DC-20) when applied to Kevlar fiber enhances the fiber tensile strength in an epoxy matrix. Inasmuch as the release agent weakens the interlaminar bond between the fiber and resin, the composite now becomes susceptible to water and solvent penetration and entrapment, which could result in the degradation of the function of the release agent and a resulting loss of composite strength. However, use of this release agent has been restricted to the hoop wrap which is by design never in direct contact with the insulation.

This minimizes the exposure of water and solvents to the hoop wraps internally, but protection must be provided on the outside.

Resin

Composite cases normally employ an anhydride or amine cured epoxy resin system, sometimes modified with plasticizers to vary the elongation, strength, and glass transition temperature characteristics. In general, these families of epoxy resins are unaffected by water, weak acids, bases, and organic solvents at room temperature conditions. Water-boil tests do indicate some immediate loss in strength, but effects are reversible and full strength is regained after drying.

Fiber Damage

Filament wound pressure vessels rely on the ability of continuous filaments to carry the pressure loads in a case. If the filaments are damaged or broken, the portion of the load carried by the broken fibers will have to be transferred in shear through the resin matrix material to the adjacent undamaged filaments. This obviously will reduce the margin of safety of the motor case depending upon the size of the case, the number, and type of composite layers affected, and the location of the damaged section.

Composite Contamination

Both fiber and resin systems are relatively insensitive to exposure to the water and solvent systems planned for use in case salvaging at room temperature conditions. Precautions would include a thorough drying of the reclaimed case structure and the limited use of acids and base constituents in the hydromining and solvent operations involving Kevlar cases.

Multiproof Testing

Reclamation of cases would require a verification of structural integrity by the performance of another proof test.

Except for a few newer case programs like C-4, there has not been a requirement to design for multiple proof testing of composite cases. Therefore, since some of the cases that are reclaimed will have been proof tested at least once, and have not been designed with a multiproof test requirement, the effects of another proof test might be a concern.

There have been studies made on the effects of a second proof cycle. The most useful work is summarized in the following references:

"The Effects of Repeated Loading on Filament Wound Pressure Vessels," by Dr. John Outwater, University of Vermont, 5 September 1963 (Defense Documentation Center No. AD422866).

"High Performance Fiber Epoxy Composite Pressure Vessels," Chiao, Hamstad, Jossop and Tolands, Lawrence Livermore Laboratory, 12 December 1978 (U.S. Dept of Commerce NTIS No. UCRL-52533).

These reports provide the following information and tentative conclusions which will be used on this program:

1. Repeated cycling will result in a reduction in the subsequent burst pressure of the pressure vessel in comparison to a vessel that is burst without preliminary cycles.

2. The fatigue life of the pressure vessels at the higher load levels (30% for glass and 90% for Kevlar) depends on both the number of stress cycles and the time at peak pressure during each cycle.

3. Composite cases are fairly noisy during pressurization. Presumably, the noises represent steps on the path to eventual burst. Acoustic emissions data showed that, after an initial cycle, there is little noise until the pressure reaches

about 90% of the previous pressure. At this time the noise level increases markedly.

4. Some reduction in burst strength occurs due to cycling. Apparently this reduction occurs fairly early in the cycle history and remains constant for a considerable number of cycles.

5. Most of this effect occurs on the first cycle with some increment on at least the second and third cycle as evidenced by the acoustic emissions recorded between 90% and 100% of the initial cycle.

6. Cases that are held at higher pressure levels for extended times degrade structurally.

From these reports, it can be assumed that if the composite material properties have not been degraded due to aging, service life conditions, or reclamation processes; the additional proof test should not significantly affect the structural integrity of the case. This assumption is based on the fact that most motor cases are proofed at pressures sufficiently below the critical levels as defined in the referenced documents, and as a result, will not be degraded below the design requirements by the additional proof test.

6.3 EFFECT ON CASE RELOADABILITY

The use of solvents, including steam to remove propellants, will have possible detrimental effects on the insulation materials. Experience with organic solvents such as tetrahydrofuran and methylene chloride for cleaning rubber parts indicate that swelling of rubber does occur, depending on the length of exposure. However, upon drying the rubber returns to its normal thickness and size.

Besides changing the apparent physical size of the rubber, it has been found that solvents will also extract plasticizers and antioxidant from the rubber compound even though it will not dissolve the cured rubber stock itself. This effect occurs locally at the surface, which is critical because of interface bonds with new insulation and liner.

The primary criterion for the selection of the solvent will, of course, be propellant dissolvability. However, the effects of the solvent on the insulation material relative to both rubber integrity and bondability must be identified. This information will be used to plan the insulation rework/removal plan for the motors undergoing solvent exposure.

The criteria that will determine the reloadability of the case will be the ability to bond to the remaining insulation and case fiber composition without subsequent adverse effects on adhesive bonds, liner bonds, and propellant liner interface bonding.

6.4 EFFECT OF FLUIDS ON COMPOSITE CASE PROPERTIES

All composites considered in this program will absorb fluids. The rate of absorption can be calculated once diffusivity is known. The rate of absorption depends upon temperature. When water is absorbed, the matrix dominated properties decrease. The amount of decrease depends upon moisture content, material type, and type of loading. There is not a clear consensus of opinion regarding recovery of properties after moisture removal. Recovery from 0 to 100% has been reported.

All composites considered for use in rocket motor cases contain organic material and will, therefore, absorb fluids contacted by the composite. Most of the literature on effect of fluids on composites discusses only the effect of water. However, reports are also available on the effects of jet fuel[1] and both polar and nonpolar solvents[2] on composite properties. The following discussion applies directly to water absorption, but the same general conclusions are also applicable to other fluids.

Testing for the effect of moisture on composites is typically completed either after exposure to high relative humidity (95% normally) or to boiling water. The effect of these two environments is the same provided that exposure times are adjusted so that the same amount of moisture is induced by each environment.[3] It is generally agreed that 2 hr of submersion in boiling water is equivalent of 1 mo of submersion in water at 72°F.[2,4]

The rate of moisture absorption can be calculated using Fick's laws.[5]

The rate of transfer of diffusing substance through unit area of a section is proportional to the concentration gradient measured normal to the section, i.e.,

$$F = -D \frac{\partial C}{\partial X} \tag{1}$$

F = the rate of transfer per unit area

C = the concentration of diffusing substance

X = distance normal to the section

D = diffusivity

The fundamental differential equation of diffusion in an isotropic medium may be derived from equation (1). If the diffusion is one-dimensional there is a gradient of concentration only along the X-axis.

$$\frac{\partial c}{\partial t} = D \frac{\partial^2 c}{\partial X^2} \tag{2}$$

Equations (1) and (2) are Fick's first and second laws of diffusion.

These equations can be used to determine the rate of moisture absorption in composites provided the orthotropic nature of diffusivity is properly accounted for.

A composite material for rocket motor case applications consists of fiber strands in a resin matrix. The moisture diffusivity depends upon the orientation of the fibers since the diffusivity along the fibers is, in general, higher than in the direction across the fibers.

The followig equations for diffusivity in the X-direction in a composite fiber were presented in Reference 6.

$$D_X = D_{11} \cos^2 \alpha + D_{22} \sin^2 \alpha \tag{3}$$

where α is the angle between the fibers and the X-axis.
D_{11} and D_{22} are the diffusivities in the directions parallel and normal to the fibers.
If D_X is not known it may be estimated from the diffusivity of the matrix D_r and the volume of the fibers v_f.

$$D_X = D_r \left[(1-v_f) \cos^2 \alpha + (1-2\sqrt{v_f/\pi}) \sin^2 \alpha \right] \tag{4}$$

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When fibers are parallel to the surface, equation (4) reduces to

$$D_X = D_{22} = (1 - 2\sqrt{\nu_f/\pi})\, D_r \qquad (5)$$

Typical diffusivity values are:

Composite	Diffusivity, ft^2/hr
Fiberglass/Epoxy	
Normal to Fibers	2.76×10^{-10}
Parallel to Fibers	1.24×10^{-7}
Kevlar/Epoxy	
Normal to Fibers	6.59×10^{-10}
Parallel to Fibers	2.36×10^{-7}

Typical moisture time profiles are given in Reference 7.

The maximum moisture content is insensitive to temperature but depends upon the moisture content in the environment.[6] For Kevlar-49/epoxy immersed in water, the saturation level is about 5% by weight.[8] The time required to reach saturation is dependent upon temperature since the diffusivity increases with temperature. Equations for maximum moisture content and time required to reach saturation are given in Reference 6.

It has been shown that moisture gradients of only 1% in adjacent plies can significantly reduce the residual strength of the composite by causing transply cracking.[9]

Various investigators[2,3,8,10-22] have observed that exposure of fiber-reinforced epoxy composites to moisture leads to a reduction in matrix dominated strength and modulus properties. The degree of strength reduction depends upon the type of failure mechanism and upon the moisture concentration. Strength reductions up to 60% (saturation, tested at 240°F) for Kevlar-49/epoxy composites[8] and up to 35% (200 hr in boiling water) for glass/epoxy composites[17] have been observed. Because of the dependence of strength degradation upon material, moisture level, exposure time and temperature,

test vehicle (degradation is different for fiber controlled and matrix controlled properties), and level of prestress,[22] it is difficult to summarize the results of the papers reviewed. Figure 6-1 from Ref 21 contains a plot relating strength loss, exposure time, and exposure temperature for glass/epoxy pressure vessels subjected to 95% relative humidity then hydroburst. The following table shows a rough time-temperature-degradation history for glass/epoxy composites subjected to a water environment:

Degradation(%)	Time (hr)	Temperature (°F)	Reference
5	168	70	19
30	36	212 (boiling)	17
35	200	212 (boiling)	17

Vaughn[17] claims that the degradation of glass/epoxy composites appears to follow a log time relationship for tensile, flexural, and compressive strength. This relationship holds only until the equilibrium moisture level is approached.[12]

Many authors[3,16,22] report that the original strength will be regained if the moisture is removed from the composite. However, other authors[4,20] report from little to 100% recovery.
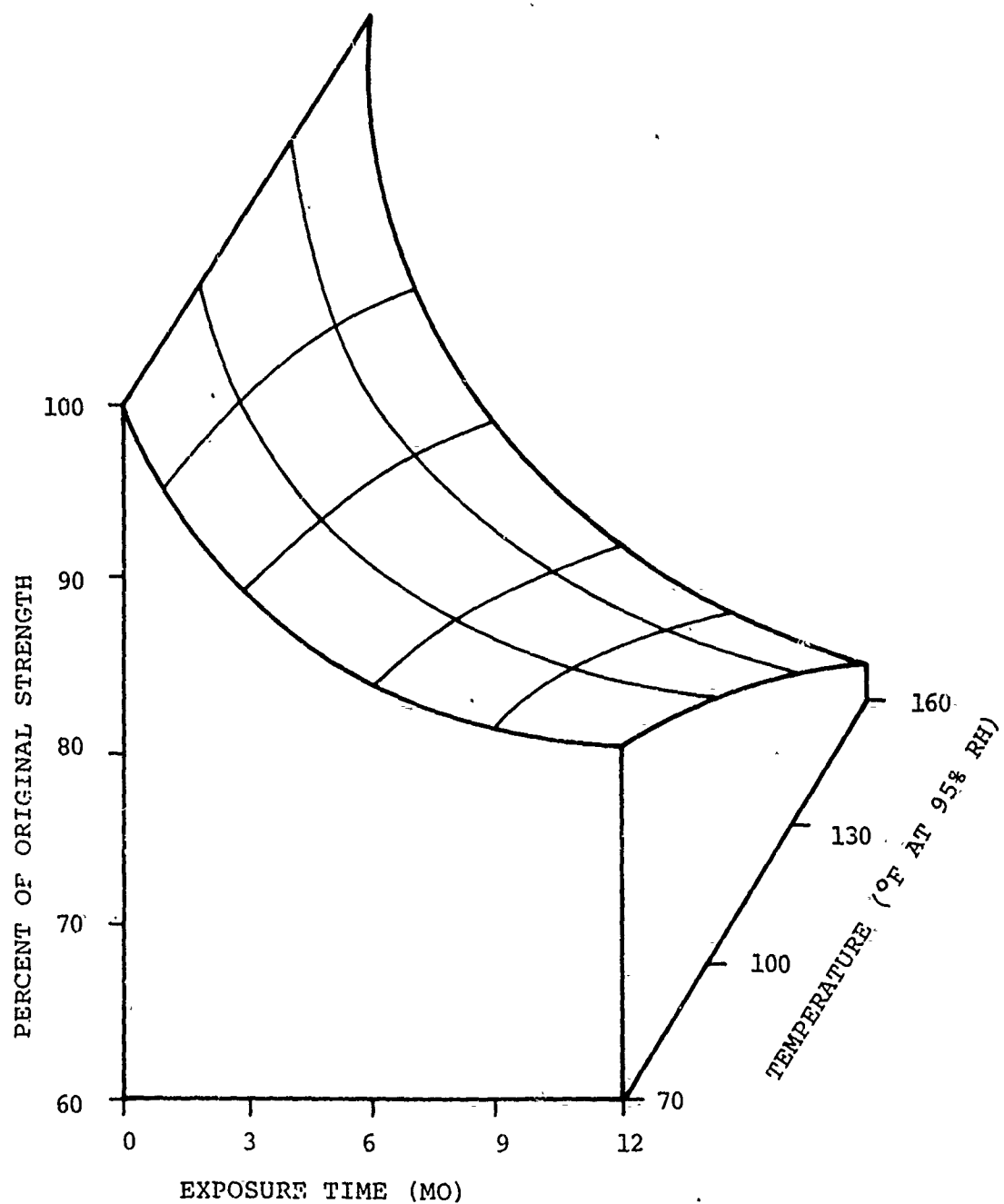
Figure 6-1. Effects of Exposure Time and Temperature
on Hydrostatic Burst Strength at 100°F of Filament
Wound Glass-Epoxy Pressure Vessels

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6.5 FRACTURE/FATIGUE AND GENERAL ACCUMULATED DAMAGE

One major problem encountered in working with fatigue in composite cases is that the types of flaws in such materials are not generally of the part-through crack type so typical of metals. Thus, in composites, a wider range of initial flaws are found than in metals. These may be a result of production process problems, such as porosity or delamination caused by contamination or poor fiber-matrix bonding caused by poor wetting of the fibers. Or they may be caused in the handling process due to impacts. In addition, the proof test itself is known to have potential for damage in the composite.[1] The physical damage itself may be basically characterized as being of three types: fiber breaks; matrix cracking or fiber-matrix debonding; and delaminations between plies. However, because all three of these types of damage often occur together for a particular flaw, characterization of a flaw is considerably more difficult than it is for metals. In addition, the criticality of each type of damage can be quite different for different types of loading. Therefore, it is not possible to define the criticality of a particular flaw in a composite through a simple device such as the stress intensity factor in metals, although the determination of an "effective" stress intensity factor may be possible.

The above differences in damage types had considerable impact on the specific details of the Fracture/Fatigue Prediction for composite SRM cases. First, it results in less reliability in finding flaws by the various NDI techniques. Thus, although delaminations can usually be spotted by existing NDI techniques, other flaws of a smaller, but perhaps more detrimental nature, such as fiber breaks, can often be missed.

Second, the determination of flaw growth rates in composites due to sustained or repeated loads is greatly complicated by the lack of anything similar to the growth of a dominant crack as is found in metals. This has led many researchers to characterize the growth of damage in composite through the

loss of residual strength or the loss of stiffness.[2] Also
the three types of damage can have substantially different
growth rates, and the rate of growth of each can be very de-
pendent on loading conditions; e.g., tension or compression.

Third, the three basic types of damage can be combined in
virtually limitless combinations in any flaw or damage region,
making it very difficult to predict a critical load for a particu-
lar physical damage region. A difference between composite mate-
rials and metals can be of great advantage. The major advantage of
importance here is that fiber dominated graphite composites
subjected to tensile-tensile fatigue are virtually indestruct-
ible, having almost flat S-N curves over a large number of
cycles.[4] Furthermore, although relatively notch sensitive
under static loading, the notch sensitivity has been observed
to decrease with initial repeated tensile loading due to the
development of a diffuse damage zone at the notch tip causing
relief of the stress concentration.[5] Instead of the inverse
relationship observed in metals, graphite composites show in-
creases in fracture toughness with increases in composite tensile
strength. However, decreases in temperature, within the operat-
ing ranges experienced by rocket motor cases, cause no notice-
able decreases in fracture toughness of composites[6] but
have detrimental effects on metals.

It has also been demonstrated that if the applied stress
level is kept below 80% of the static strength, glass fiber
reinforced materials exhibit very little loss in strength in
low cycle application.[7]

These differences in material behavior also have a sub-
stantial impact on the specific details of design. Specifi-
cally, although the determination of initial flaw size, growth
under sustained or repeated loads, and flaw criticality is more
difficult for composites than for metals, it appears that in
the case of tension loading of fiber dominated cases, these
determinations may be unnecessary. That is, because of the

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flat S-N curves exhibited by composite materials and the tendency of flaws to become less critical in the initial stages of repeated loading, it appears that a proof test of the composite case to any load above the operating loads will insure the success of the mission.

The effects of damage in composites have been assessed by many researchers. This research however has been primarily restricted to the area of impact damage. References 7 to 11 are typical of this work. As a general conclusion, however, the results of these studies, both theoretical and experimental, are specifically oriented with respect to fiber type and layup matrix material, and application that it is difficult to draw general conclusions from the work. It does appear that graphite fiber matrix is much less tolerant to damage than S glass, but that as a class composite materials do not tend to be overly tolerant to damage.[11]

Also the exact type and orientation of the damage are major factors involved in the assessment. There are devices that tend to make a filament material more tolerant to damage but again no generalization can be drawn.

Many studies report excellent correlation between theoretical damage predictions and experimental results as it relates to broken fiber and matrix damage.[12]

It appears that low cycle fatigue as it relates to a low number to test cycles should not be a major problem in the salvage of composite cases. Incidental damage however must either be prevented or evaluated based on test programs designed and directed to specific fiber, matrix, and applications.

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6.6 SEPARATION OF BUNA-N INSULATION FROM FIBERGLASS/EPOXY COMPOSITE CASES

During the Minuteman Long Range Service Life Analysis Program, 50 case/insulation samples were cut from the barrel portion of an aged third stage Minuteman case. Each specimen was dried for 8 hr in a 135°F vacuum chamber, followed by conditioning at 135°F, 80% RH for 10 to 132 days. At various

conditioning times, samples were removed from conditioning and the rubber peeled from the fiberglass.

The bond was very good for samples tested early in the program. We were able to peel the insulation from the case samples. However, fibers were occasionally damaged.

After 132 days conditioning, the first fiberglass ply was removed with the insulation by applying very little force, indicating severe degradation of the interlaminar bond strength.

Thus in the process of case reclamation, extreme caution must be exercised to prevent normal and bending loads from being applied after moisture has penetrated into the case.

The following literature was reviewed for composite case degradation information.

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APPENDIX B

PHASE II INTERIM REPORT

FEASIBILITY AND COST STUDIES

CONTENTS

ILLUSTRATIONS

TABLES

PHASE II - FEASIBILITY AND COST STUDY

DEVELOPMENT OF COMPOSITE CASE SALVAGE PROCEDURES

INTRODUCTION

The proposal to reclaim composite cases from rocket motors represents an advancement in the state-of-the-art. Reclamation of steel cases from defective or decommissioned solid propellant motors for reloading has proven to be cost effective without degrading case reliability. By adapting or utilizing methods developed for steel case reclamation, a limited number of composite cases have been salvaged and reused during development programs.

The current program, Development of Composite Case Salvage Procedures, consists of a four-phase effort to determine and verify methods which can be used to salvage composite cases. Phase I consisted of making a technological survey of existing and potential processing methods which could be applied to case salvage. Phase II consists of determining the cost effectiveness of the different methods and making an initial assessment of feasibility. Phase III consists of conducting laboratory studies to evaluate the processing methods and verify or disprove initial assessments. Phase IV consists of developing a program plan, utilizing full scale motors, to verify the methods selected in the previous phases.

OBJECTIVE

The objective of Phase II was to develop a method of comparison between salvage techniques and to develop cost models for comparing the costs of manufacturing new cases versus the cost of salvaging cases from existing motors.

SCOPE

The goal was to computerize a model that predicted salvage case costs and new case fabrication costs for comparison. To do this it was necessary to establish cost equations and determine the drivers which affect the costs. Built into this model were the following parameters:

1. Learning curve adjustments.
2. Production drivers such as quantities, rates and schedule.
3. Propellant sensitivities.
4. Motor size (4,000 to 200,000 lb range).

5. Inspection and testing costs.

6. Facilities and tooling costs.

In addition, a computer program was needed to evaluate or tabulate the assessed risk of the selected process methods. The risk assessment included potential hazard to facilities and personnel, potential damage to the insulation and case, and an evaluation of reloadability, complexity of operations, and whether the method is proven or untried.

RESULTS

1. Tabulation and regression analysis of fabrication costs yielded a model for prediction and comparison of new case costs.

2. A cost model was developed and structured to predict, combine and trade costs for composite case salvage techniques.

3. Trade studies were conducted on a cross section of motors to determine cost effectiveness in salvaging different motor sizes.

4. A computer program was completed for tabulating the assessment of risk for different salvage operations.

CONCLUSIONS

1. Salvage of composite cases from motors containing Class 1.3 propellant appears to be feasible.

2. The cost effectiveness of case salvage is dependent upon:
   a. The age of the motor.
   b. Motor size.
   c. Complexity of the case design.
   d. Case quantity requirements.
   e. Does a new case production line need to be maintained?
   f. What processing losses from case salvage can be tolerated?
   g. Salvage facility and tooling availability.
   h. Qualification requirements.

3. Salvage of composite cases from motors with Class 1.1 propellant does not appear to be cost effective based on current information.

4. The best methods for removal, based upon current evaluation of estimated costs and assigned risk values, are:
   a. Propellant removal by hydromining or machining.

b. Liner removal by low pressure hydromining, steam or mechanical abrasion.

c. Insulation removal by heat and peal method or, for glass only, low pressure hydromining.

5. Salvage of flaps appears to be impractical due to the ease with which they can be damaged.

6. Complete removal of the insulation does not appear to be practical, due to the potential of damage to the case.

7. This study advances the state-of-the-art for evaluation of whether potential case salvage operations are cost effective.

8. Propellant ingredient recovery for resale or reuse, especially when large quantities of the same propellant are available, appears to be cost effective.

RECOMMENDATIONS

1. Long term effects of salvage processes on reclaimed cases should be conducted.

2. No significant changes should be made in the Phase III laboratory efforts. The laboratory studies in Phase III should emphasize testing to evaluate (a) removal of Class 1.1 propellant, (b) the effects of solvents upon the case and insulation, and (c) the potential for damage during removal of the insulation.

3. The values assigned for the assessment of risk should be reevaluated after the studies of Phase III are completed.

4. In-depth studies to reclaim propellant ingredients should be conducted.
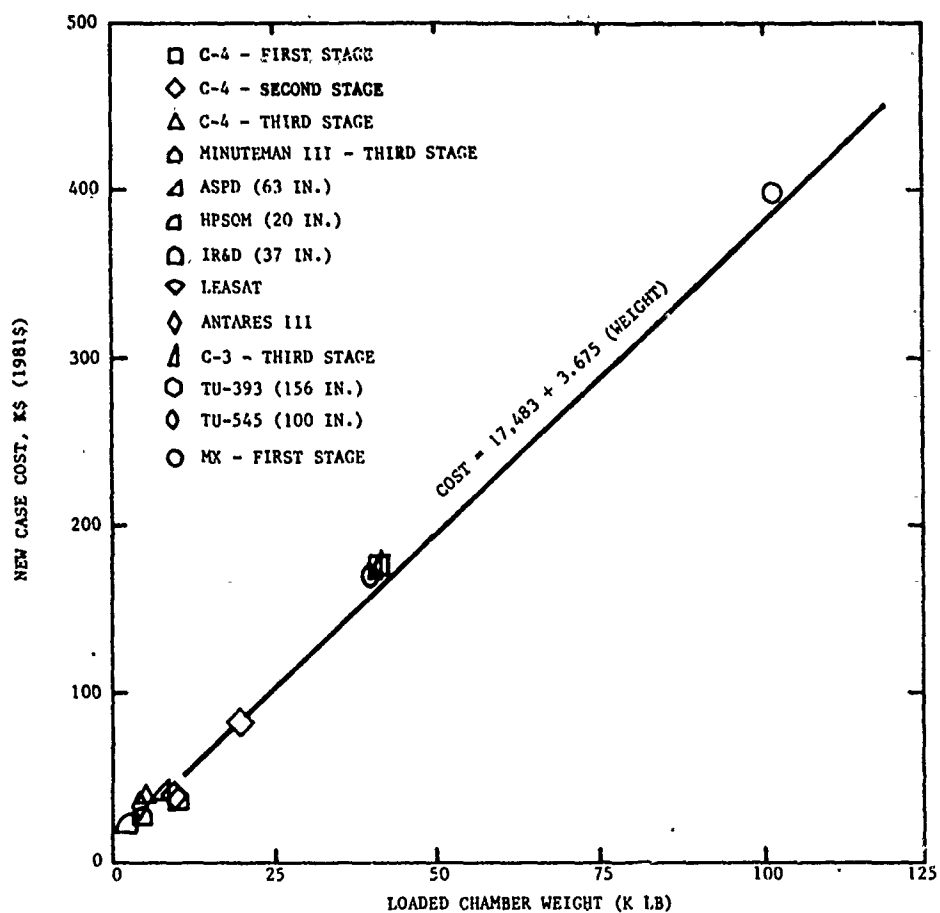
Figure 1. New Case Cost (Labor, Material and Support) vs Loaded Chamber Weight

DISCUSSION

A. Development of the New Case Cost Model

Cost histories on fabrication of new cases were compiled and a regression analysis was conducted to determine the parameters important to cost data correlation. It was determined that the primary parameter was the loaded grain weight; i.e., the chamber, insulation and propellant weights combined. Hence, the cost of the labor and materials, CLM, can be estimated by:

$$CLM = 17483 + 3.6746 \text{ (loaded grain weight)}$$

The weight of the insulation correlated well with the equation:

$$\text{Weight of Insulation} = 0.0277 \text{ (propellant weight)}^{0.9277}$$

The amortized costs of the facilities and the equipment can then be added to the above labor and materials cost to obtain an estimated manufacturing cost for a new case fabrication. Because facilities and tooling requirements and costs vary from company to company, most of the comparisons have been performed excluding facility and tooling costs.

A plot of the data used and the resultant regression curve is shown in Figure 1. Deviation from the model can be attributed to complexity factors. Cases with TT ports, multiple nozzles or space motors designed for high pressure operations cost more than the norm.

B. Development of Case Salvage Costs

The basic method of development of the cost model was to break the salvaging process into its various steps and to sum the effect of each step to obtain the total cost for a particular salvage operation. The tasks involved in a salvage operation have been divided as follows:

1. Handling cost, CH, the receipt of the case and initial receiving inspection.

   $$CH = f \text{ (motor size and propellant class)}$$

2. Bulk propellant removal costs, CBPR,

   $$CBPR = f \text{ (method of removal, cutting rate, motor size)}$$

   Where the cutting rate $= f$ (propellant sensitivity and physical properties)

3. Residual propellant removal costs, CRPR:

   CRPR = f (method of removal, cutting rate, motor size)

4. Liner removal costs, CLR:

   CLR = f (propellant sensitivity, surface area, method of removal)

5. Insulation removal costs (from the cylindrical section), CIC:

   CIC = f (method of removal, propellant sensitivity and surface area)

   NOTE: An additional factor is added if the the case was embedded and/or if it had TT ports.

6. Insulation removal costs (from the dome areas) CID:

   CID = f (method of removal, propellant sensitivity and surface area)

7. In-process inspection, CI:

   CI = f (level of salvage, surface area)

8. Reinsulation costs, CRI:

   CRI = f (motor size and complexity of design)

9. Final inspection and qualification costs, CIQ:

   CIQ = f (methods used [i.e., X-ray, hydrotest and qualification] motor size and propellant class)

10. Waste disposal costs, CWD:

    CWD = f (method of disposal, class of propellant, motor size)

The options that can be selected for each computation are as follows:

1. Level of salvage.

2. Methods of removal for:

   a. Bulk propellant.

   b. Residual propellant.

   c. Liner.

   d. Insulation, cylindrical section.

   e. Insulation, dome section.

3. Level of final inspection.

4. Method of waste disposal.

5. Facilities and equipment requirements.

At each computation of the cost of a task, the option can be made as to whether facilities and equipment costs are to be included. The options available are: (1) no facilities and/or equipment to be included, (2) a set standard facility and/or equipment cost for a specific method of removal,

or (3) the actual facility and/or equipment cost may be used, if available.
If the method of removal for a process step is the same as previously has
been used, the calculation automatically eliminated the addition of facilities
and equipment for the next step. This allows for comparison of the type of
facility that would be most cost effective. For example, a permanent hydro-
mining facility was used to reclaim steel Minuteman Third Stage cases. The
increased risk in hydromining Class 1.1 propellant indicates the desirability
of building a very minimal cost facility with remote operations and acceptance
of the risk of possible replacement of the facility and equipment. The result
could be that the original facility cost for Class 1.1 propellant would be
less than for Class 1.3.

The rate of production affects the cost computation in two ways. The propellant
removal rate includes a labor cost for setup, cutting and cleanup periods. If
the production rate is too high for a single facility to handle, then a second
facility is automatically added. The second manner for production rate to
affect the cost is that at higher production rates, effective use of labor and
material increases and the percentage for program overhead decreases; both
factors reduce the cost. Relationships developed from composite case fabri-
cation have been used to estimate the reduced production cost as the rate
increases. Similarly, a learning curve has been employed to reduce the
average cost for labor as the number of units to be processed increases.

The levels of salvage are: (1) removal of propellant, (2) removal of propel-
lant and liner, (3) removal of propellant, liner and insulation in the cylin-
drical section, and (4) removal of propellant, liner and all insulation.
This differs slightly from the proposal conditions. The judgment made during
Phase I was that it would be necessary to remove and replace the flaps in all
salvage operations. Experience also indicated, at that time, that removal
of insulation from the cylindrical section may be feasible, whereas complete
removal of insulation from the dome was very unlikely.

The optional methods considered for bulk propellant removal are: (1) high
pressure hydromining, (2) low pressure, hot water hydromining, (3) wet
machining, (4) dry machining, (5) chemical degradation with hydromining
(low pressure), (6) chemical degradation with machining, and (7) burnout.

TABLE I
SUMMARY OF COMPARISON OF MINUTEMAN III RESULTS
WITH CASE SALVAGE COST ESTIMATIONS

(MINUTEMAN III GLASS CASE: PROPELLANT WEIGHT = 7298 POUNDS;
AREA = 38.7 FEET^2; CLASS 1.3 PROPELLANT; SINGLE MOTOR SALVAGE
OPERATION)

TASK	MINUTEMAN III[1]	ESTIMATED SALVAGE COST[2]	
SALVAGE LEVEL	4	4	2
METHOD OF REMOVAL			
A. PROPELLANT, BULK	12680	9917	9917
B. PROPELLANT, RESIDUAL		2606	2606
C. LINER	2923	-	2478
D. INSULATION, CYLINDRICAL	3553	2779	-
E. INSULATION, DOME		1040	-
RECEIVING AND HANDLING	420	645	323
INSPECTION - IN-PROCESS	0	4807	3965
INSPECTION - FINAL	2895[3]	6761	6761
QUALIFICATION PROGRAM	0	0	0
REINSTALLATION OF INSULATION	4591	5143	5143
WASTE DISPOSAL	0	722	722
COST PER UNIT, (1981$)			
@ 1 PER MONTH		33800	31900
NEW CASE COST, (1981$)			
@ 1 PER MONTH		45800	45800
DIFFERENCE (NEW - SALVAGE), (1981$)			
@ 1 PER MONTH		12000	13900

NOTES:
1. COSTS BASED ON ACTUAL MANHOURS CHARGED.
2. INCLUDES ESTIMATED LABOR, MATERIALS AND SUPPORT..

3. INCLUDES X-RAY ONLY; DOES NOT INCLUDE HYDROTEST.

The options available for residual propellant removal are: (1) all propellant removed by the bulk removal method, (2) hydromining, low pressure, hot water, (3) wet machining, (4) dry machining, (5) chemical degradation with hydromining, (6) chemical degradation with machining, and (7) burnout.

The options available for liner removal are (1) liner removed with residual propellant, (2) hydromining, low pressure, hot water, (3) chemical dissolution, (4) mechanical [i.e., buffing, grinding, etc], and (5) steaming.

The options available for insulation removal, either from the cylindrical or dome sections, are: (1) same as previously used methods, (2) hydromining, low pressure, hot water, (3) chemical soak with hydromining, (4) chemical soak with mechanical removal, (5) mechanical removal [i.e., buffing, grinding, etc], (6) manual buffing, and (7) heat and peel.

Options of the inspection level are: (1) visual and dimensional inspection during the processing steps, (2) X-ray inspection of the chamber and re-installed insulation, (3) hydrotest including hydroburst of a specific number of cases, and (4) loading and firing of a specific number of reconditioned cases for qualification.

The options for waste disposal are: (1) open pit incineration, (2) closed incineration [i.e., rotary kiln, fluid bed, etc), (3) use as explosive and (4) reclamation of solid ingredients for reuse.

The constants and functions used in the equations for computing the costs were developed from cost data from various ongoing programs at Thiokol. The validity of the final results was checked by comparison of computed costs with the actual costs recorded during the Minuteman III Case Reclamation Program. This comparison (Table I) demonstrates that costs computed for the individual tasks compare favorably with the actual costs. Zero costs were accounted for in the Minuteman III program for waste disposal and in-process inspection because these functions were treated as support costs.

The methods of salvage corresponded to the methods used in the Minuteman III tests, which were:

1. Propellant removal by hydromining.
2. Liner removal by buffing, grinding.
3. Insulation removal by hydromining.

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The computed examples selected used the same method where applicable. In a Level 4 salvage, it is assumed that the liner is removed with the propellant or insulation, not as a separate step; therefore, no cost is tabulated. In a Level 2 salvage, no insulation is removed, therefore, these costs are omitted in the calculation. The insulation cost is probably high, since only repair and replacement of the flaps would be required.

In addition to obtaining actual costs for specific operations, the following results were obtained from the Minuteman III program which were beneficial to this program.

1. Reclamation of the flaps was determined to be impractical. Attempts to salvage the flaps were unsuccessful.

2. Operating conditions needed for removal of insulation by hydromining were defined; however, results indicated a high risk of damage to the case fibers if insulation is removed by this method.

3. The heat and peel method was confirmed to be practical for removing insulation from the cylindrical section of the case; however, removal of the bonding adhesive from the case was difficult.

4. Application of low pressure steam proved to be very effective for removing the liner from the insulation.

5. Tests conducted on Kevlar case sections indicated that severe delamination occurs due to water damage when insulation is removed by hydromining.

It was concluded that there was a need to define the hydromining operating conditions for propellant removal that would minimize the potential damage to the insulation. This work was planned for Phase III.

It is evident that the combinations of examples that can be calculated are almost limitless. Figure 2 summarizes the results of calculations for various methods of salvage of Minuteman III cases. These results indicate that the level of salvage and the method selected for propellant removal have the most effect on the total cost for a specific motor.

The effect of the motor size is shown in Figure 3, which shows how the cost of the salvage increases as the motor size increases for a motor containing Class 1.3 propellant.
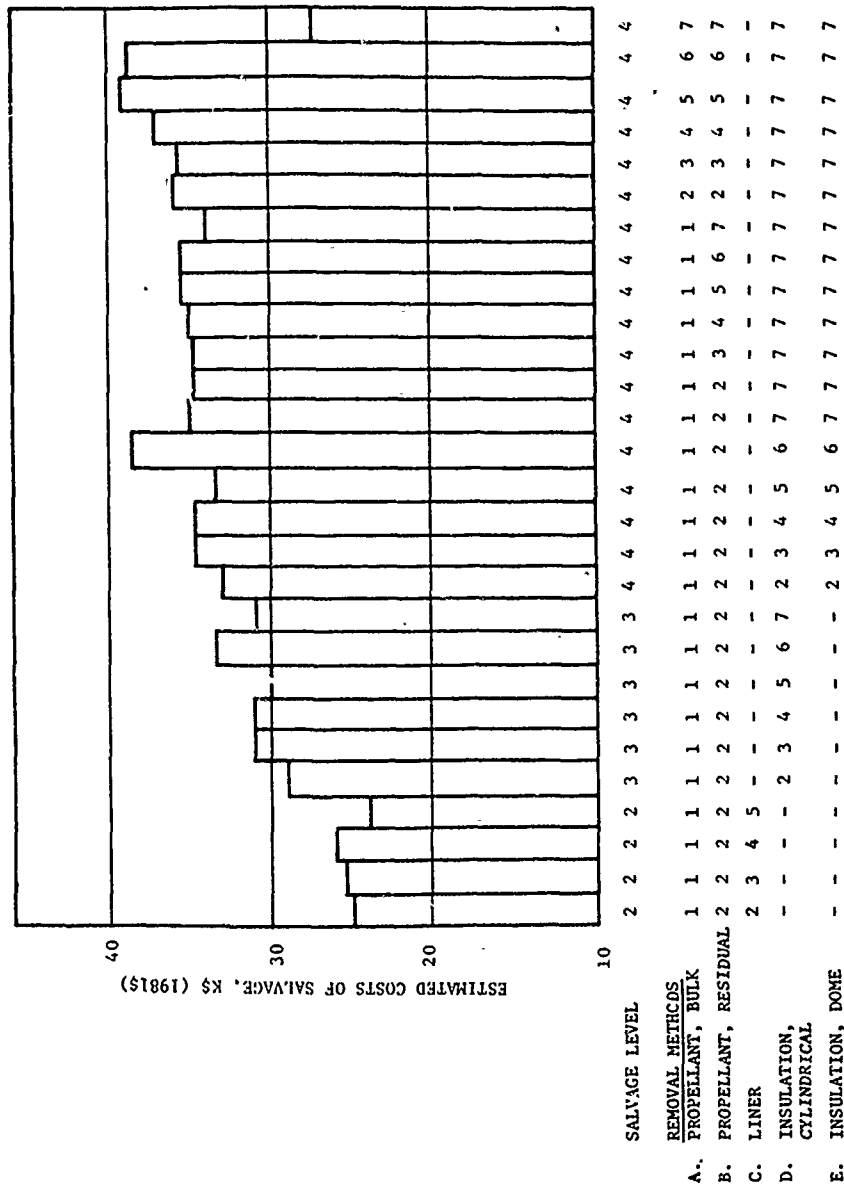
ESTIMATED COSTS OF SALVAGE, K$ (1981$)

40   30   20   10

SALVAGE LEVEL

REMOVAL METHODS
A. PROPELLANT, BULK
B. PROPELLANT, RESIDUAL
C. LINER
D. INSULATION, CYLINDRICAL
E. INSULATION, DOME

Figure 2. Summary of Estimated Costs of Salvage for Various Methods of Removal and Salvage Levels for a Minuteman III Size Case

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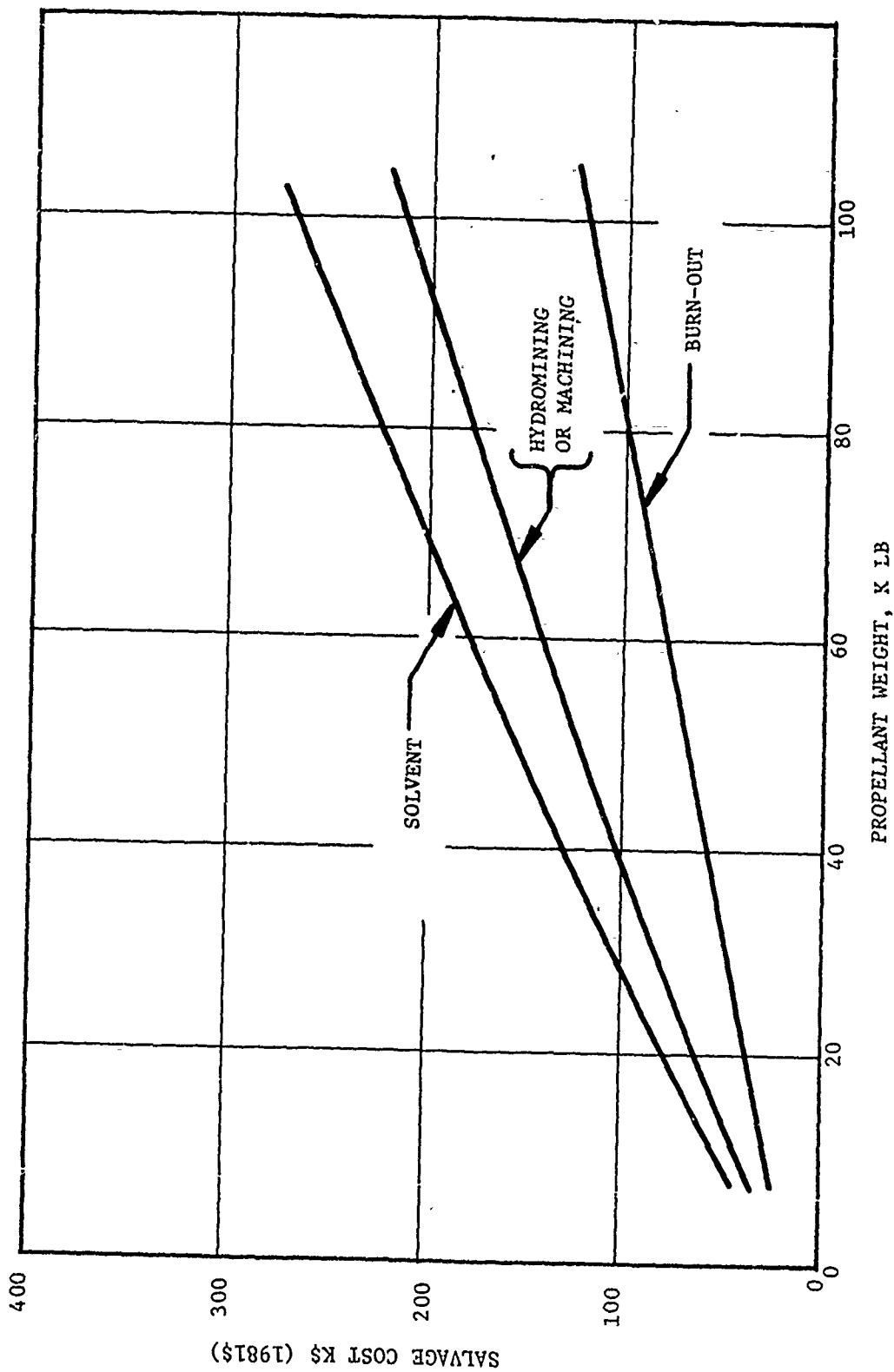
Figure 3. Approximate range of salvage costs for cases with
Class 1.3 propellant as a function of motor size (propellant weight)
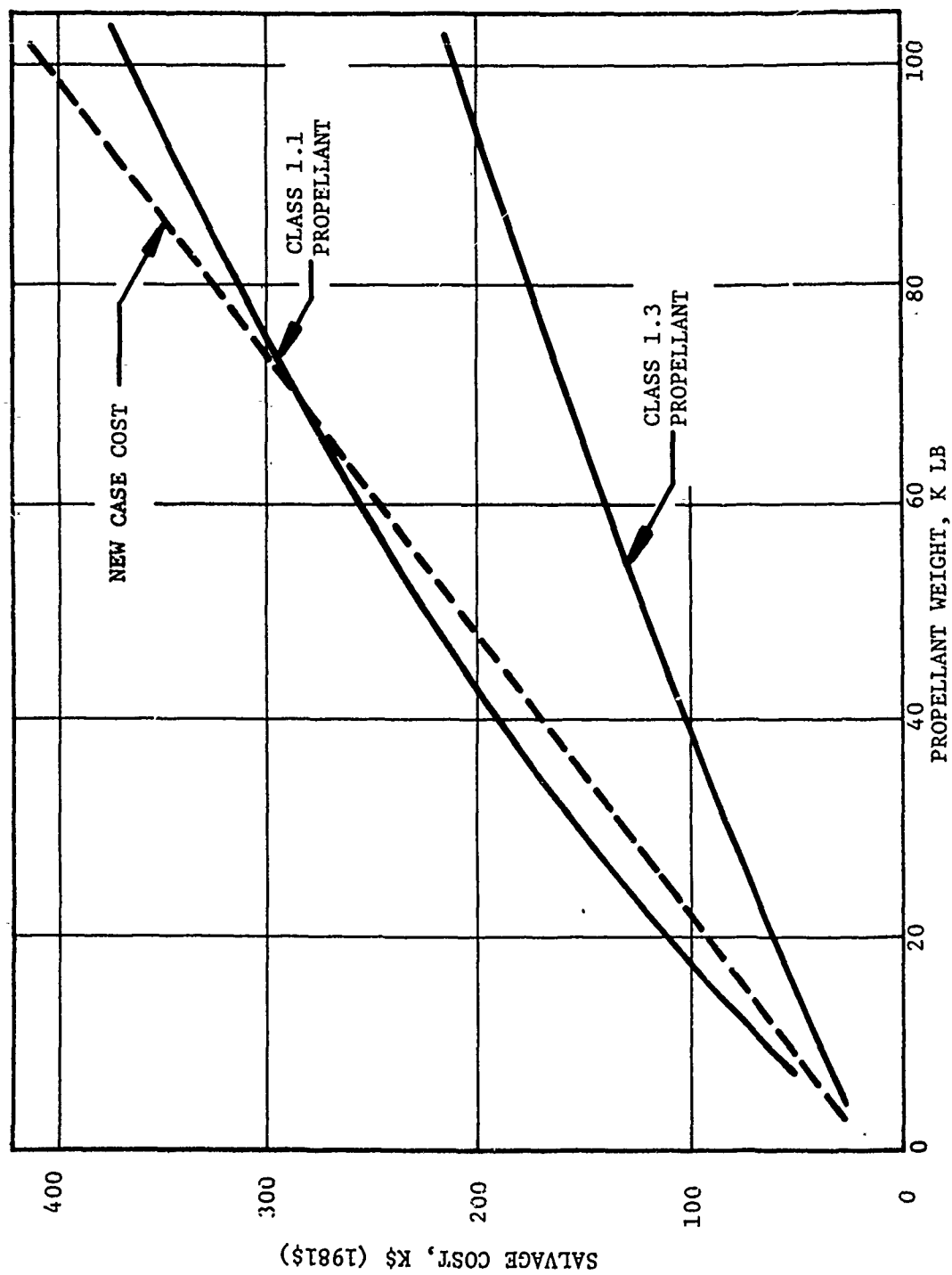
Figure 4. Comparison of new case cost with salvage costs for chambers with Class 1.3 and Class 1.1 propellants

OUTLINE OF CASE SALVAGE PROCESSES (

Propellant	Manufacturer (Origin)	Type of Case	Special Facilities Requirement	Method of Bulk Propellant Removal	Method of Removing Residual Propellant	L Meth
ANB-3066	Aerojet Minuteman III, Stage III	S Glass	Remote cutting Corrosion Prevention	Hydromine to approximately 3 in. from insulation.	Use hot water hydromining	Meth Epox Poss polyn moin
TP-H1207	Thiokol Proposed for MX Stage I	Kevlar 49	Remote cutting Corrosion prevention Friction level indicates cautions necessary to prevent ignition (such as low pressure on hydromining and/or slow speed for mechanical cutting).	Hydromine to 3 in. from insulation	Unknown at present. Preliminary testing indicates solvent such as tetrahydrofuran may soften binder system sufficiently to soft wash with hydromining or hand cut to insulation. Water - steam would leach AP and reduce hazard to personnel. Or check and test hydromining with 150°F water at 1000 psi.	Limi minn Low requi sligh (com see A ignlti frictl indic theri used
TP-H1202	Thiokol Proposed for IFSM-II	Kevlar 49	Same as TP-H107	Same as TP-H1207	Same as TP-H1207	Same
CYH (DDP)	Hercules Inc. Minuteman II, Stage III	S Glass	Facilities exposed to nitrate ester systems will have to meet the critical design requirements for these materials which would include sealed surfaces, impact prevention, friction prevention, and a total hazards analysis for the operation	Mechanical cutting utilizing tools design to cut the propellant to a predetermined configuration would be utilized. The cutting speed, depth of cut, type, and amount of coolant used on the cutting blade would be determined. Waste and chips would be removed periodically to minimize any build-up of subdivided material that could cause detonation to deflagration.	The remaining film of propellant would be removed by first solvent treatment, either irrigation or vapor dispersion to partially remove the nitroglycerin and to solvate and degrade the binder to whatever limit necessary to utilize hand cutting or soft hydromining (sufficient pressure to cut the remainder of the propellant without cutting the rubber). Warm water hydro-mining is considered to be effective in removing the remaining propellant from the insulation.	The i the b be ut facili prote glyce prod. pros waste cuttin resid migh woul small in a l mate this r facili comp and p proba hydra to pr appos
VRP	Hercules Inc. & Thiokol Joint Venture C-4 Trident also (similar to MX-Stage III)	Kevlar 49				
TP-N1023	Thiokol	Kevlar 49				

TABLE II

ROCESSES CHOSEN FOR CANDIDATE PROPELLANT SYSTEMS

Logic for Method Selected	Method for Removal of Insulation	Logic for Method Selected
Method used to remove HB polymer. Epoxy cured propellant from Poseidon fiberglass case – HC polymer is susceptive to moisture degradation.	Heat and peel thin insulation in cylindrical section. Hot water hydromining will remove the remaining cement. Grinding will be used in the dome region to remove excess, degraded insulation.	With application of heat to soften the cement/adhesive the insulation is easily peel from the cylindrical section. Hot water hydromining (150°F at 3000 psi) has proven effective to remove the cement. The risk is considered to be too great to completely remove the insulation in the dome, especially near the metal polar bosses.
Limited testing indicates hydromining cuts the propellant. Low pressure hydromining may be required because propellant is slightly friction sensitive (comparison to other types see ANb 3066 – also SRAM had ignition on washout and it was friction sensitive). Literature search indicates HTPB is degraded by – OH; therfore, caustic addition to water used in hydromining may be beneficial.	Mechanical grinding or buffing will be used for insulation removed. Complete removal is considered a poor risk.	Heat application, either by hot water or heat gun, to remove insulation exposes the surface fibers in Kevlar cases and produces case damage. Solvent soaking, including water, is not recommended.
Same as TP-H1207	Same as TP-H1207	Same as TP-H1207
The mechanical cutting to remove the bulk of the propellant would be utilized to minimize the facility investment necessary to protect a facility from nitroglycerin contaminated waste product streams that would be present with hydromining. The waste products for mechanical cutting such as the chips and the residual coolant material which might contain nitroglycerin would be removed batchwise in small quantities and destroyed in a burning area. The hazardous materials could be contained in this manner. Tooling design and facility layouts would require complete safety hazards analysis and provide a safety incident probability to 10^{-6}. Warm water hydromining accomplished at 500 – 100 psi to provide a soft impact on the system, appears to be feasible.	Heat and peel insulation in cylindrical sections. Hot water hydromining to remove cement. Mild buffing or hand scraping of insulation to remove age degraded or propellant contaminated (NG) surfaces. Mild mechanical grinding or buffing will be the primary candidate method. Same as VRP	Hot water hydromining receptable for glass cases. Due to probable NG contamination the buffing of insulation must be mild. Water or solvents may be needed to desensitize the surface if NG is present. Heat application increases the risk to the Kevlar case. The use of solvents to aid removal is also prohibited due to their effect on the Kevlar fibers. Same as VRP

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TABLE III

EVALUATION OF MOTOR SALVAGING TECHNIQUE

Explosive Class	Method	Advantages	Disadvantages
1.3	Hydromining	• Moderate raw material cost • Facilities already exist • Low tooling and equipment cost • Low personnel safety risk • Low facility safety risk • Effective, fast removal • Successfully applied in previous programs • No effect on reloadability	• High potential to damage insulation • High potential to damage case • Large quantities of contaminated water to treat
1.1	Hydromining	• Moderate raw material cost • Cost of equipment moderate • Low personnel safety risk; remote operation • Effective, fast removal • No effect on reloadability	• New facilities required • New tooling and equipment • Moderate facility risk • High potential to damage insulation • High potential to damage case • Large quantities of contaminated water • Application resulted in ignition and loss of facility
1.3	Low-pressure, high-temperature hydromining	• Low raw material cost • Facilities exist • Low - moderate equipment cost • Low personnel safety risk • Low to moderate facilities safety risk • Low potential to damage insulation • Low potential to damage case • Moderate amount of water contamination • Effective to use • Successfully applied in previous programs • No effect on reloadability	• High energy cost to heat water

TABLE III

EVALUATION OF MOTOR SALVAGING TECHNIQUE (CONT)

Explosive Class	Method	Advantages	Disadvantages
1.1	Low Pressure Hydromining	. Low raw material cost . Facilities already exist . Low - moderate equipment cost . Low personnel safety risk; remote . Low potential damage of insulation . Low potential damage of case . Moderate amount of water contaminated . Effective removal . Successfully applied in previous programs . No effect on reloadability	. High energy cost to heat water . Moderate to high facilities hazard risk . Water contaminated with NG
1.3	Machining	. Low raw material cost . Moderate facility cost . Moderate equipment cost - part already existing . Low personnel safety risk . No potential to damage case . Low potential to damage insulation . Small amount of water contaminated . Successfully used in similar applications . No effect on reloadability	. Moderate facility safety risk . Potential to recover water solubles . Relatively slow for removal of bulk
1.1	Machining	. Low raw material cost . Moderate facility cost . Low personnel safety risk . Low potential to damage insulation . Low potential to damage case . Small amount of water contaminated . Successfully used in similar application . No effect on reloadability	. Moderate facility safety risk . Relatively slow removal . High equipment and tooling cost

TABLE III

EVALUATION OF MOTOR SALVAGING TECHNIQUE (CONT)

Explosive Class	Method	Advantages	Disadvantages
1.3	Solvent Degradation	Low to moderate facility safety risk Improved rate of removal May decrease explosive hazard by desensitization of propellant	High raw materials cost High facility cost (including secondary removal method) High equipment and tooling cost High waste disposal cost High personnel safety risk (solvent toxicity) Moderate potential to damage insulation Moderate potential to damage case New, untried method Potential effect on reloadability
1.1	Solvent Degradation	Decrease facility risk factor Improved rate of removal Decreased explosive hazard by desensitization of propellant	High raw material cost High facility cost (must include secondary removal method) High equipment and tooling cost High waste disposal cost High personnel safety risk (solvent toxicity) Moderate potential to damage insulation Moderate potential to damage case New, untried method Potential effect on reloadability
1.3	Burn Out	Low raw material cost Low facility cost Low equipment and tooling cost Low waste disposal cost Effective, fast removal	Moderate personnel safety risk High potential to damage insulation High potential to damage case Moderate to high facility safety risk Useful only for removal of residual propellant Unsuccessfully tried with steel case reclamation Potential adverse effect on reloadability

Estimation of the difference of the salvage costs between motors having Class 1.3 and 1.1 propellants is shown in Figure 4. The predicted cost of new cases is also shown for comparison. These results indicate that salvage of motors containing Class 1.1 propellants may be economically feasible only for larger cases. These estimates were based upon comparison of increased costs which occur in other steps of rocket motor manufacture such as ingredient preparation, mixing and casting, which result from extra precautions needed with Class 1.1 propellants. As more data become available, it may be found that these costs have been estimated higher than was necessary.

C. Development of Risk Assessment

Development of a computer program to evaluate the different methods of salvage proceeded as follows:

Table II indicates the chosen processes to salvage composite rocket motor cases containing the candidate propellant selected for this program. In the development of Table II, Table III was utilized. This table evaluates each of the propellant removal techniques from the standpoint of the advantages and disadvantages of the process and its relationship to the explosive classification of the propellant. Evaluation of the propellant removal techniques includes hydromining, high pressure-low temperature water hydromining, machining, solvent degradation and propellant burnout.

The insulation removal techniques were evaluated for the advantages and disadvantages that exist as applied to both "S" glass and Kevlar 49 composite case materials. The type of propellant also was considered. The processes that were selected are the heat and peel, solvent soak and peel, grinding-machining, low pressure-hot water hydromining developed by Thiokol in the Minuteman Stage III Composite Case Reclamation Program, and manual removal by buffing or scraping.

The propellant disposal techniques also were evaluated for their advantages and disadvantages based upon the type of propellant disposal techniques and include open pit incineration, closed incineration [i.e., rotary kiln incinerators, fluidized bed incinerators, etc], reclamation of ingredients from the propellants, and waste propellant used directly as an explosive for commercial industry.

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The criteria for methods selection, listed in Table IV, are cost, person-
nel safety and facility risk, potential damage to the insulation and case,
reloadability of the case after reclamation, the effectiveness of the
process and the confidence factor based upon the history and success that the
method has experienced to its current state of development. In each method,
the rating factor proceeds from 0 to 10. In all cases, the rating factors
indicate that a more desirable or feasible process would have a lower number
rating than the other methods to which it is compared. For example, a
rating of 0 represents a low cost or low risk estimate for the process.

TABLE IV

CRITERIA FOR SELECTION OF METHOD

Parameters Considered	Rating Factor		
	Low	Moderate	High
Safety, Risk to Personnel	0	5	10
Safety, Risk to Facilities	0	5	10
Potential Damage to Insulation	0	5	10
Potential Damage to Case	0	5	10
Reloadability Difficulty	0	5	10
Effectiveness**	0	5	10
Confidence Factor***	0	5	10

** Effectiveness is based upon rate of removal and ability to remove all
   by one method

*** Confidence is based on previous history of success of method for the
    same or similar operation

Tables V, VI and VII summarize the analyses of the various processes for
propellant removal, insulation removal and the propellant disposal methods,
respectively. Each parameter from Table IV was judiciously applied to each
process in Tables V thru VII, culminating in a scoring method that indicates
the desirability of each process. The total tabulation scoring for each
process was then made and these results were reflected in the choices of the
proposed salvaging techniques listed in Table II. It is evident that not
all possibilities have been investigated by the development of these prelimi-
nary selection criteria. Improvement of the selection criteria will occur

as data are obtained from laboratory studies during Phase III.

In each example in Table II, propellant removal, insulation removal and propellant disposal methods have been used to provide a total summation of the salvage operation. The insulation replacement method was not so rigorously treated. Current studies on insulation replacement indicate that the vulcanization and precuring of the insulator on outside molds will be required. The preformed insulator segments will then be secondarily bonded into the salvaged case in the same manner used to install internal insulation systems in current rocket motor production.

*Solvent soak must be followed by another means of removing the propellant such as machining or hydromining.

Propellant Class	Method of Operation	Cost, Raw Material	Cost, Facilities	Cost, Equipment	Cost, Waste Disposal	Safety, Risk to Personnel	Safety, Risk to Facilities	Potential Damage to Insulation	Potential Damage to Case	Reloadability Difficulty	Effective	Confidence Factor	Total
1.3	Hydromining	5	1	3	7	0	2	6	2	3	0	0	29
1.1	Hydromining	5	5	5	9	2	7	6	2	3	0	10	54
1.3	Hot Water, LP, Hydromining	8	1	4	4	0	0	2	2	2	7	0	30
1.1	Hot Water, LP, Hydromining	8	5	6	6	2	5	2	2	2	2	6	46
1.3	Machining, Wet	1	3	4	3	2	1	7	0	1	8	0	30
1.1	Machining, Wet	1	6	8	5	4	6	7	0	1	8	1	47
1.3	Machining, Dry	0	4	4	1	7	8	5	0	0	10	2	41
1.1	Machining, Dry	0	8	8	2	9	10	5	0	0	10	4	56
1.3	Solvent Soak*	10	8	8	8	8	4	8	8	8	10	6	86
1.1	Solvent Soak*	10	10	10	10	4	8	8	8	8	10	6	98
1.3	Burn Out	1	2	1	0	4	5	10	10	10	1	8	52
1.1	Burn Out	1	4	2	1	6	9	10	10	10	1	10	64

Parameters Considered in Selection

TABLE VI

BASIS FOR SELECTION OF INSULATION REMOVAL METHOD

Propellant Class	Case	Method of Operation	Cost, Raw Material	Cost, Facilities	Cost, Equipment	Cost, Waste Disposal	Safety, Risk to Personnel	Safety, Risk to Facilities	Potential Damage to Insulation	Potential Damage to Case	Reloadability Difficulty	Effective	Confidence Factor	Total
1.3	Glass	Heat and Peel	0	1	4	1	2	0	NA	2	2	0	0	12
1.3	Kevlar	Heat and Peel	0	1	4	1	2	0	NA	2	2	0	0	12
1.1	Glass	Heat and Peel	0	5	7	5	5	5	NA	2	2	0	0	31
1.1	Kevlar	Heat and Peel	0	5	7	5	5	5	NA	2	2	0	0	31
1.3	Glass	Solvent Soak & Peel	5	4	5	5	8	0	NA	6	10	0	8	51
1.3	Kevlar	Solvent Soak & Peel	5	4	5	5	8	0	NA	8	10	0	8	53
1.1	Glass	Solvent Soak & Peel	5	8	8	10	10	5	NA	6	10	0	8	70
1.1	Kevlar	Solvent Soak & Peel	5	8	8	10	10	5	NA	8	10	0	8	72
1.3	Glass	Grinding, Mechanical	0	3	6	1	2	0	NA	6	0	4	2	24
1.3	Kevlar	Grinding, Mechanical	0	3	6	1	2	0	NA	6	0	4	2	24
1.1	Glass	Grinding, Mechanical	0	8	10	5	5	5	NA	6	0	4	2	45
1.1	Kevlar	Grinding, Mechanical	0	8	10	5	5	5	NA	6	0	4	2	45
1.3	Glass	Hydromining	1	5	5	4	0	0	NA	6	8	2	2	33
1.3	Kevlar	Hydromining	1	5	5	4	0	0	NA	10	8	2	2	37
1.1	Glass	Hydromining	1	10	7	8	5	5	NA	6	8	2	2	52
1.1	Kevlar	Hydromining	1	10	7	8	5	5	NA	10	8	2	2	56
1.3	Glass	Hand Scraping	0	0	0	1	2	0	NA	4	0	10	8	25
1.3	Kevlar	Hand Scraping	0	0	0	1	2	0	NA	4	0	10	8	25
1.1	Glass	Hand Scraping	0	5	1	5	8	5	NA	4	0	10	8	46
1.1	Kevlar	Hand Scraping	0	5	1	5	8	5	NA	4	0	10	8	46

Parameters Considered in Selection

TABLE VII

BASIS FOR SELECTION OF WASTE DISPOSAL METHOD

Propellant Class	Method of Operation	Parameters Considered in Selection											
		Cost, Raw Material	Cost, Facilities	Cost, Equipment	Cost, Waste Disposal	Safety, Risk to Personnel	Safety, Risk to Facilities	Potential Damage to Insulation	Potential Damage to Case	Reloadability Difficulty	Effective	Confidence Factor	Total
1.3	Open Pit Incineration	0	0	0	N/A	1	0	N/A	N/A	N/A	0	0	1
1.1	Open Pit Incineration	0	0	0	N/A	2	0	N/A	N/A	N/A	0	0	2
1.3	Closed Incineration	0	7	7	N/A	2	0	N/A	N/A	N/A	5	2	28
1.1	Closed Incineration	0	10	10	N/A	3	10	N/A	N/A	N/A	5	2	40
1.3	Use as Explosives	5	5	5	N/A	5	5	N/A	N/A	N/A	2	10	37
1.1	Use as Explosives	0	8	8	N/A	8	10	N/A	N/A	N/A	2	8	44
1.3	Reclaim Ingredients	0	7	8	N/A	8	5	N/A	N/A	N/A	2	8	38
1.1	Reclaim Ingredients	5	10	10	N/A	10	10	N/A	N/A	N/A	2	10	57

APPENDIX C

PHASE III INTERIM REPORT

LABORATORY STUDIES

R&D STATUS REPORT

DEVELOPMENT OF
COMPOSITE CASE SALVAGE
PROCEDURES

SUBMITTED TO

DEPARTMENT OF THE AIR FORCE
HEADQUARTERS, AIR FORCE FLIGHT TEST CENTER
DIRECTORATE OF CONTRACTING (PKRA)
EDWARDS AFB, CA 93523

In Accordance With
Contract FO4611-81-C-0001
CDRL No. 1

December 1981

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DEVELOPMENT OF COMPOSITE CASE SALVAGE PROCEDURES

AFRPL Contract F04611-81-C0001

1.0 INTRODUCTION

This program is an 18-month effort, divided into four phases, to develop
safe, cost effective methods to remove Class 1.1 and 1.3 propellants from
solid rocket motors that have composite cases. Phase I consisted of a
technical assessment of current recovery methods and included a literature
search for information applicable to the salvage processes. Phase II
consisted of a feasibility and cost study during which cost parameters
and models were established for cost comparisons between reclaimed and new
cases. Phase III consists of laboratory testing of propellant, liner and
insulation removal methods deemed promising from the results of the Phase I
and Phase II efforts. Phase IV will consist of outlining a qualification
program to demonstrate the salvaging techniques selected in the previous
phases utilizing three government furnished Minuteman III third stage motors.

2.0 OBJECTIVE

This report summarizes the rsults of all work efforts conducted during
Phase III of the AFRPL Composite Case Salvage Program. Phase III is the
laboratory studies designed to evaluate potentially cost effective and/or
feasible methods for propellant, liner or insulation removal as were identified
in Phases I and II. A special emphasis has been placed on determining the
potential risks involved in the processes, particularly the risks concerned
with handling Class 1.1 propellant and with utilization of solvents to
aid propellant and/or liner removal.

3.0 SUMMARY

Tests were conducted to evaluate or to obtain processing data on the more
promising propellant removal methods: hydromining, machining, burn-out
and solvent degradation. The data indicate that hydromining and machining
are the better methods with lower risk factors.

The effect of the propellant removal methods on the insulation and case materials were evaluated to assess the risk for potential damage to the reclaimed case. Particular emphasis was placed on the effects of solvents upon the insulation and case materials and the effect the solvents may have on the reprocessing step required to prepare the case for reloading with propellant. The high potential to damage (risk) associated with the solvents indicates their utilization is unfeasible. It has also be concluded that there is some risk in utilizing hydromining to remove the last of the propellant. Utilization of hot water and low pressures during hydromining reduces the risk.

4.0   CONCLUSIONS

The conclusions of the tests and evaluation performed during Phase III are as follows.

1.   Hydromining is a viable method of propellant removal for all propellants, both Class 1.1 and Class 1.3 propellants. Proper conditions can be tailored .o each propellant to minimize the potential for ignition.

2.   Reduction of water temperature and pressure during hydromining greatly reduces the potential for damage of the insulation and/or case.

3.   Dry machining could be an effective removal method but the increased safety factor resulting from water flooding during wet machining effectively eliminates the utilization of dry machining.

4.   The assessment of high potential for case damage eliminates the burn-out method from further consideration despite its apparent economic advantages.

5.   Several solvents were found which degrade the different propellants; however, most of the solvents that degraded the propellant were deleterious to the case, particularly to the resin. If they were strong enough to attack the propellant binder system, they also attacked the case resin system.

6.   Solvent effects on the insulation itself appeared to be transitory; however, many solvents migrated through the insulation sufficiently to weaken the insulation/case bond and attack the resin system of the case.

7. The accumulated data provide a good basis for planning the follow-on program.

5.0   RECOMMENDATIONS

It is recommeded that the results of these evaluations be accepted as fulfillment of the required work for Phase III of Contract F04611-81-C-0001, following the planned oral review. It is recommended that approval be granted for the continuation of the program, Phase IV effort.

6.0   DISCUSSION

The objective of Phase III was to evaluate the processing techniques identified as feasible during Phase I and Phase II through laboratory-type operations. At the conclusion of Phases I and II, it was concluded that hydromining and machining were the apparent best methods for propellant removal. Economically the burn-out method, burning the propellant from the case at lower pressures, was promising but the risk of damage to the case was judged to be high. Due to the potential economic benefit, additional evaluation was deemed necessary. Utilization of solvents to degrade the propellant was judged to be more expensive than any of the other methods, due to the solvent costs plus the increased cost of handling the solvents and risk was also expected to be high.

Two basic considerations indicated the need to further evaluate the use of solvents. The one industrial example of hydromining Class 1.1 propellant resulted in an incident of ignition and damage to the plant.[1] Hydromining of composite Class 1.3 propellants has been shown to be technically feasible and a state-of-the-art operation. Thus, the major area of interest was the development of safe methods for removal of Class 1.1 propellants. Degradation of the polymer or solids dissolution to weaken the propellant or to desensitize it and facilitate machining or hydromining was a worthwhile objective.

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[1]Bingham, J. F., et. al., "Removal of CDB Propellant from Case Bonded Rocket Motors by High Pressure Water Jet," IMI Ltd., Summerfield Research Station, Kidderminster, G. B.

The other consideration was concerned with the disposal of the propellant. Continued disposal of propellant by incineration, especially open-pit incineration, is regarded as wasteful and harmful to the environment. Recovery of the propellant ingredients is seriously being considered. If the degradation of the propellant and/or the dissolution of the solids is the first step in an ingredient recovery scheme, then the overall process incorporating both case reclamation and propellant ingredient recovery may be favorable economically.

Most of the effort in Phase III, therefore, was expended in tests to further evaluate the propellant removal methods and to determine the associated risk of damage to the insulation and/or the case. Evaluation of insulation removal methods and reinsulation of the case was limited since the investigations previously concluded during the Thiokol third stage Minuteman III salvage tests were considered to be applicable and definitive.

The results of the third stage Minuteman III indicated that the cases could be economically salvaged with minimum damage to the internal insulation. The risk of case damage involved with removal of the internal insulation and replacement was too great to warrant consideration with aged motors. Case salvage is most feasible where the motor is not aged and the insulation may remain intact.

6.1 PROPELLANT REMOVAL METHODS

6.1.1 Hydromining

The primary objectives of the hydromining tests were accomplished. The results obtained were as follows.

1. Cutting rates were measured and correlation between propellant properties was obtained.

2. Hot-water, low-pressure hydromining appeared to be to be beneficial. Cutting effectiveness was increased for scme propellant. Other pro-pellant hot water hydromining may have little or no beneficial effect.

3. Class 1.1 propellant was tested at conditions which were felt to be extreme and no ignition was evident. These tests were not conclusive. Though indicative that class 1.1 propellants can be hydromined, additional testing of each particular propellant would be required to determine under what conditions ignition may occur.

A total of 128 tests were conducted on propellant samples. Three nozzles having throat diameters of 0.055, 0.085 and 0.125 inches were used. The smaller 0.055 inch nozzle has an elongated converging section which produces a very fine, pencil-lead-type spray for several feet. The samples were placed two inches from the nozzle exit. The test results have been grouped by propellant in Table I. The propellants are identified in Section B of Table I. The normal procedure was to increase the pressure incrementally at a given water temperature until the water cut through the four-inch thick propellant sample. Each test consisted of a sweep period when the nozzle was rotated onto the propellant and a dwell period after the nozzle came to rest with water impinging on the propellant loaf carton. During the high pressure (10,000 psi) and hot water (190°F) impact tests, a steel plate was placed behind the carton to increase the severity of the test. The testing of the CYH propellant was limited to the high-pressure, hot-water impact conditions due to the limited supply of CYH propellant.

Figures 1 through 6 show the test facility and equipment used in the impact (hydromining) tests. Figure 1 shows the remote control bunker with the air flow valve and electrical switch in the entryway. The water blaster, con-sisting of a reservior, high-pressure water pump and a diesel engine to drive

TABLE I. PROPELLANT/HIGH-PRESSURE WATER IMPACT TESTS RESULTS

| Propellant | Nozzle Diameter, Inches | Test # | Water Conditions | | | Speed* Inch/Second | Sweep Cut | | Time,* Seconds | Dwell Cut | |
			Temperature °F	Pressure psi	Velocity Feet/Sec		Depth, Inches	Area/Time Inch²/Second		Depth, Inches	Depth/Time Inches/Second
TP-H1202	.125	98	65	2000	383	0.1	1	0.10	30	3½	0.17
		99	65	3000	460	0.1	½	0.05	21	Through	>0.19
		100	100	3000	460	1.3	¼	0.33	20	Through	>0.20
		101	150	2000	383	1.3	1/8	0.16	16	3	>0.19
		102	150	1000	255	0.8	1/8	0.10	30	3	0.10
		103	170	1000	255	1.0	1/8	0.13	40	3	0.08
		104	170	2000	383	1.3	1/8	0.16	30	Through	>0.13
		105	170	3000	460	1.3	½	0.33	8	Through	>0.50
TP-H1202	.055	80	70	3800	659	.4	½	0.20	30	2	0.07
		81	70	5000	791	.7	3/4	0.58	30	3	0.10
		82	70	6000	850	.6	3/4	0.45	30	3½	0.17
		83	70	7000	922	.7	1½	1.05	9	Through	>0.44
		84	100	5000	791	.4	3/4	0.30	30	3½	0.11
		85	100	6000	850	.4	1½	0.50	6	Through	>0.67
		86	100	7000	922	.7	2½	1.75	8	Through	>0.50
		87	150	6000	850	.5	1½	0.63	14	Through	>0.29
		88	150	5000	791	.4	1½	0.60	24	Through	>0.17
		89	170	4000	720	.4	½	0.20	36	Through	>0.11

* Values in parenthesis are estimated based upon time measurements made during dry runs.

TABLE I (CONTINUED)

PROPELLANT/HIGH-PRESSURE WATER IMPACT TESTS RESULTS

Propellant	Nozzle Diameter, Inches	Test #	Water Conditions			Sweep Cut			Dwell Cut		
			Temperature °F	Pressure psi	Velocity Feet/Sec	Speed * Inch/Second	Depth, Inches	Area/Time Inch²/Second	Time, * Seconds	Depth, Inches	Depth/Time Inches/Second
TP-H1202	.085	90	65	4000	440	.6	½	0.60	20	Through	> 0.20
		91	65	3000	380	.5	3/8	0.32	30	3	≥ 0.10
		92	100	3000	380	.4	½	0.10	24	Through 2½	> 0.17
		93	100	1900	276	.5	½	0.13	30	2	≥ 0.08
		94	105	1900	276	.4	3/8	0.15	30		0.07
		95	150	3000	380	.3	7/8	0.26	18	Through 2½	≥ 0.22
		96	170	1900	276	.4	½	0.20	30	2½	≥ 0.08
		97	170	3000	380	.3	2½	0.75	30	Through	≥ 0.13
		148	190+	10000	828	.8	Through	≥ 3.2	10	Through	≥ 0.40

* Values in parenthesis are estimated based upon time measurements made during dry runs.

TABLE I (CONTINUED)

PROPELLANT/HIGH-PRESSURE WATER IMPACT TESTS RESULTS

Propellant	Nozzle Diameter, Inches	Test #	Water Conditions			Speed Inch/Second	Sweep Cut		Time,* Seconds	Dwell Cut	
			Temperature °F	Pressure psi	Velocity Feet/Sec		Depth, Inches	Area/Time Inch²/Second		Depth, Inches	Depth/Time Inches/Second
ANB-3066	.125	106	65	1000	255	.7	1/8	0.09	30	2½	0.08
		107	65	2000	383	.6	1/8	0.08	30	Through	> 0.13
		108	100	1000	255	.6	¼	0.15	30	3	> 0.10
		109	100	2000	383	.7	½	0.35	15	Through	> 0.27
		110	150	1000	255	.7	¼	0.18	12	Through	> 0.33
		111	150	500	179	.8	1/8	0.10	30	2	0.07
		112	170	500	179	.8	¼	0.20	27	Through	0.15
ANB-3066	.085	113	65	1700	276	.4	1/8	0.05	25	Through	> 0.14
		147	190	10000	828	1.3	Through	> 5.2	10	Through	> 0.40
ANB-3066	.055	114	65	3800	659	.8	¼	0.20	28	Through	> 0.14

* Values in parenthesis are estimated based upon time measurements made during dry runs.

TABLE I (CONTINUED)

PROPELLANT/HIGH-PRESSURE WATER IMPACT TESTS RESULTS

Propellant	Nozzle Diameter, Inches	Test #	Water Conditions			Sweep Cut			Dwell Cut		
			Temperature °F	Pressure psi	Velocity Feet/Sec	Speed* Inch/Second	Depth, Inches	Area/Time Inch²/Second	Time,* Seconds	Depth, Inches	Depth/Time Inches/Second
TP-H1207	.085	7	60	2000	476	(.25)	1	0.25	(45)	2¼	0.05
		8	60	5000	496	(.25)	Through	1.60	(45)	Through	> 0.09
		12	60	10000	828	(.25)	½	0.13	(45)	2	-0.04
		13	60	10000	828	(.25)	¼	0.01	(45)	3	0.07
		43	65	1800	276	(.4)	1/8	0.05	(30)	2	0.07
		46	100	1800	276	(.4)	¼	0.10	(30)	2 1/8	0.07
		49	150	1800	276	(.4)	0	0	(30)	2	0.07
		52	190+	1800	276	(.4)	1/8	0.05	(30)	2¼	0.08
		57	65	4000	386	.8	¼	0.20	25	Through	> 0.16
		58	65	6000	552	.8	1	0.81	9	Through	> 0.44
		59	100	4000	386	.8	½	0.4	8	Through	> 0.50
		149	190+	10000	828	.8	Through	> 3.2	10	Through	> 0.40
TP-H1207	.055	1	60	3800	659	(.25)	Through	1.60	(45)	Through	> 0.09
		2	60	5000	791	(.25)	¼	0.13	(45)	3	0.07
		42	65	3800	659	(.4)	¼	0.10	(30)	3	0.10
		47	100	3800	659	(.4)	3/4	0.30	(30)	3¼	0.11
		48	150	3800	659	(.4)	1	0.40	(30)	Through	> 0.13
		53	190+	3800	659	(.4)	¼	0.10	(30)	Through	> 0.13
		54	65	5000	791	(.4)	1½	0.60	(30)	2 3/4	0.09
		55	65	7000	922	1.3	½	0.67	10	Through	> 0.40
		56	100	5000	791	.8	¼	0.20	23	Through	> 0.17

* Values in parenthesis are estimated based upon time measurements made during dry runs.

TABLE I (CONTINUED)

PROPELLANT/HIGH-PRESSURE WATER IMPACT TESTS RESULTS

Propellant	Nozzle Diameter, Inches	Test #	Water Conditions			Sweep Cut			Dwell Cut		
			Temperature °F	Pressure psi	Velocity Feet/Sec	Speed * Inch/Second	Depth, Inches	Area/Time inch²/Second	Time, * Seconds	Depth, Inches	Depth/Time Inches/Second
TP-H1207	.125	3	60	500	229	(.25)	1/8	0.03	(45)	1 1/8	0.03
		4	.60	1000	255	(.25)	¼	0.06	(45)	1¼	0.03
		6	60	4000	540	(.25)	Through	1.00	(45)	Through	≥ 0.09
		44	75	500	229	(.4)	¼	1.0	(30)	3/8	0.01
		45	100	500	229	(.4)	0	0	(30)	1	0.03
		50	150	500	229	(.4)	0	0	(30)	3½	0.08
		51	190+	500	229	(.4)	0	0	(30)	3	0.10
		60	65	1000	255	.5	0	0	30	1¼	0.04
		61	65	2000	383	.8	1/8	0.10	30	2 3/4	0.09
		62	65	3000	475	.7	¼	0.18	30	3¼	0.11
		63	65	4000	510	.7	1	0.70	10	Through	≥ 0.40
		64	100	500	229	.5	0	0	30	¼	0.01
		65	100	1000	255	.7	1/8	0.09	30	2	0.07
		66	100	2000	370	.7	1/8	0.09	30	Through	≥ 0.13
		67	100	3000	460	(.7)	1	0.70	24	Through	≥ 0.20

* Values in parenthesis are estimated based upon time measurements made during dry runs.

TABLE I (CONTINUED)

PROPELLANT/HIGH-PRESSURE WATER IMPACT TESTS RESULTS

Propellant	Nozzle Diameter, Inches	Test #	Water Conditions			Sweep Cut			Dwell Cut		
			Temperature °F	Pressure psi	Velocity Feet/Sec	Speed* Inch/Second	Depth, Inches	Area/Time Inch²/Second	Time* Seconds	Depth, Inches	Depth/Time Inches/Second
CYH	.035	151	195+	10000	828	.8	1 3/4	1.40	10	Through	≥ 0.40
VRP	.125	17	60	2000	360	(.25)	½	0.13	(45)	1 3/8	0.03
		18	60	4000	525	(.25)	Through	≥1.00	(45)	Through	≥0.09
		68	65	500	179	(.7)	0	0	(20)	1	0.05
		69	65	1000	280	(.7)	0	0	(20)	3/8	0.02
		70	65	2000	383	(.7)	½	0.18	(20)	1½	0.08
		71	65	3000	460	(.7)	3/4	0.53	(20)	Through	≥0.20
		72	100	500	179	(.7)	0	0	(20)	1/8	0.01
		73	100	2000	383	(.7)	½	0.18	(20)	2	0.10
		74	100	3000	460	.4	2	0.80	13.5	Through	≥0.30
VRP	.085	75	100	200	270	(.4)	½	0.50	(20)	1 3/4	0.09
		76	150	2000	270	(.4)	½	0.30	(20)	1½	0.06
		77	150	3000	386	.3	2½	0.75	30	3½	0.12
		78	150	4000	450	.5	1½	0.63	10	Through	≥0.4
		146	190+	10000	828	1.3	Through	≥3.2	10	Through	≥0.4
VRP	.055	19	60	4000	720	(.25)	1½	.31	(45)	2 3/4	0.06
		20	60	6000	860	(.25)	Through	≥1.0	(45)	Through	≥0.09
		79	65	3800	659	.4	1½	0.6	30	3	0.10

* Values in parenthesis are estimated based upon time measurements made during dry runs.

TABLE I (CONTINUED)

PROPELLANT/HIGH-PRESSURE WATER IMPACT TESTS RESULTS

Propellant	Nozzle Diameter, Inches	Test #	Water Conditions			Speed* Inch/Second	Sweep Cut		Dwell Cut		
			Temperature °F	Pressure psi	Velocity Feet/Sec		Depth, Inches	Area/Time Inch²/Second	Time,* Seconds	Depth, Inches	Depth/Time Inches/Second
TP-N1035	.125	25	62	2000	383	(.25)	2 1/8	0.53	(45)	3½	0.07
		27	100	500	179	(.25)	3/4	0.19	(45)	1	0.02
		28	100	1000	255	(.25)	3/4	0.19	(45)	2	0.04
		29	100	2000	383	(.25)	2 3/4	0.69	(45)	3 1/8	0.07
		37	100	2000	255	(.25)	Through	> 1.0	(45)	Through	> 0.09
		38	173	500	179	(.25)	0	0	(45)	1 3/4	0.04
		39	170	1000	255	(.25)	2½	0.63	(45)	2½	0.06
		40	170	2000	383	(.25)	½	0.13	(45)	3 1/8	0.07
		41	195+	1000	255	(.25)	Through	> 1.0	(45)	Through	> 0.09
TP-N1035	.085	30	100	2000	276	(.25)	1 1/8	0.28	(45)	2 3/4	0.06
		31	100	5000	480	(.25)	Through	> 1.00	(45)	Through	> 0.09
		32	100	3000	386	(.25)	2	0.50	(45)	Through	> 0.09
		33	100	2500	340	(.25)	1½	0.38	(45)	3½	0.08
		34	170	2000	276	(.25)	2½	0.63	(45)	Through	> 0.09
		35	173	2500	340	(.25)	Through	> 1.0	(45)	Through	> 0.09
		150	190+	10000	828	.8	Through	> 3.2	10	Through	0.40
TP-N1035	.055	21	60	4000	660	(.25)	2½	0.63	(45)	Through	> 0.09
		22	60	6000	850	(.25)	Through	> 1.00	(45)	Through	> 0.09
		36	100	4000	660	(.25)	Through	> 1.00	(45)	Through	> 0.09

* Values in parenthesis are estimated based upon time measurements made during dry runs.

FIGURE 1. CONTROL BUNKER USED DURING WATER IMPACT TESTS
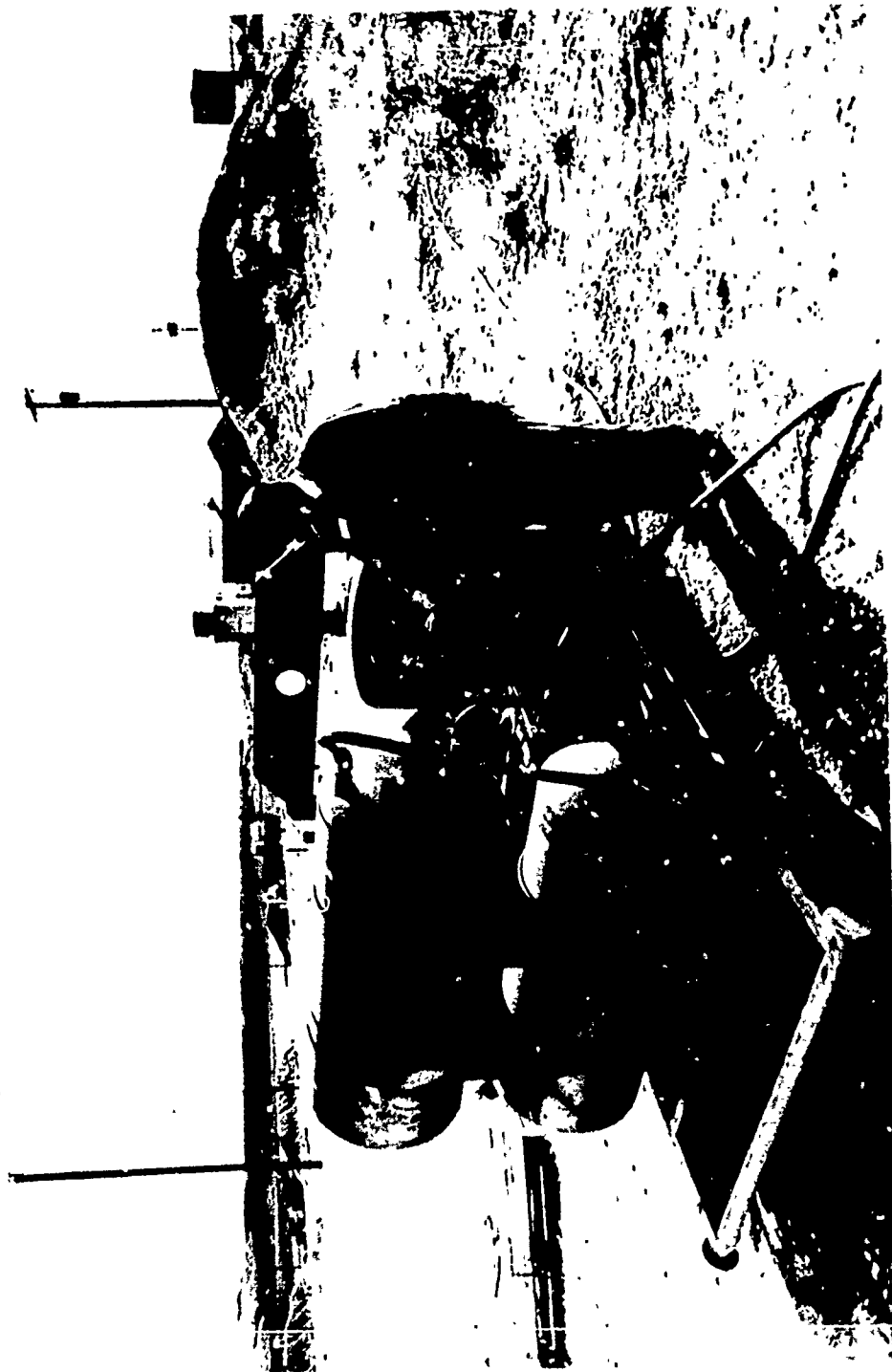
FIGURE 2. AMERICAN WATER BLASTER - HIGH PRESSURE WATER PUMP AND DIESEL ENGINE
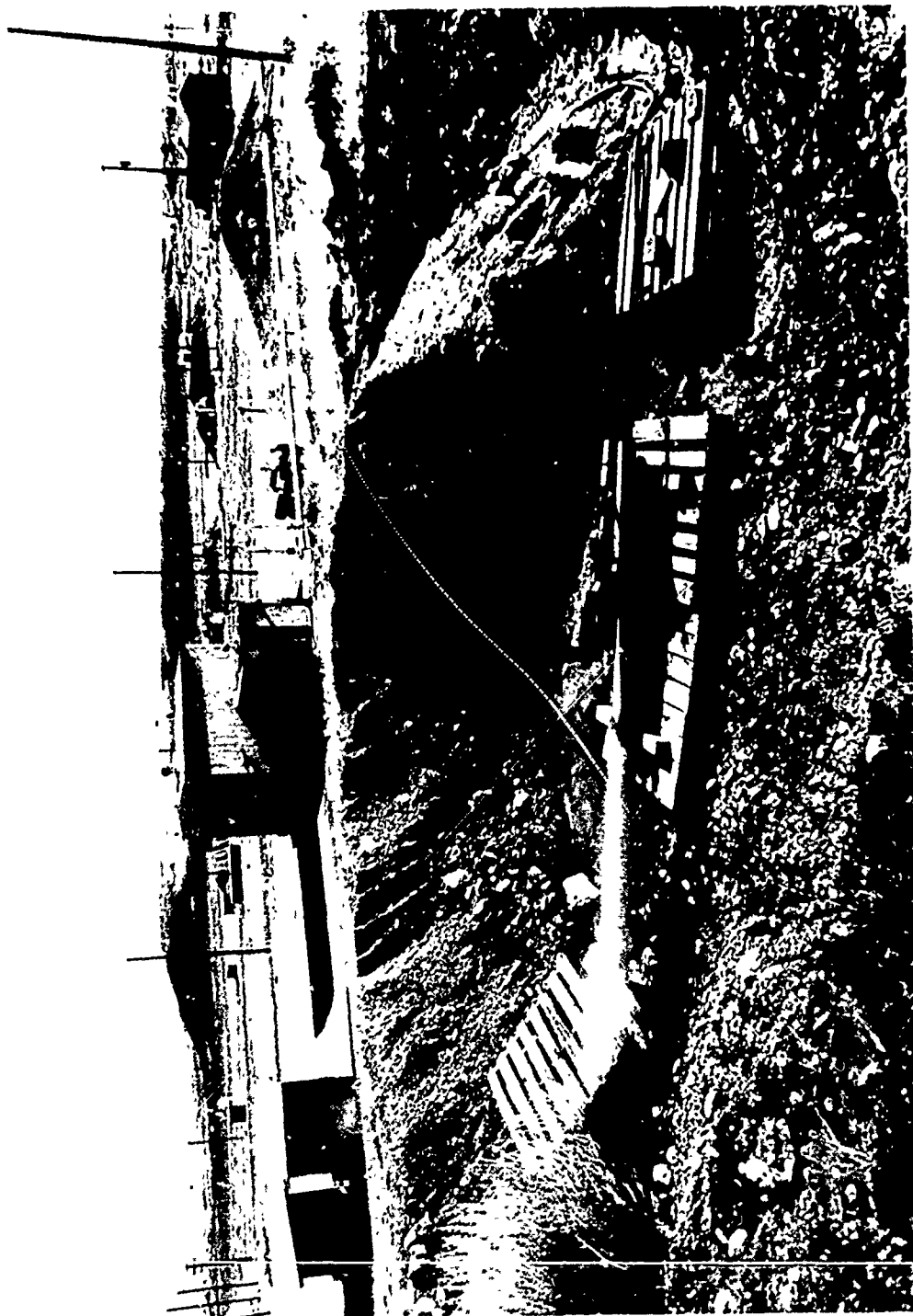
FIGURE 3. HIGH PRESSURE WATER FLOWING PRIOR TO SWINGING INTO TARGET FOR AN IMPACT TEST

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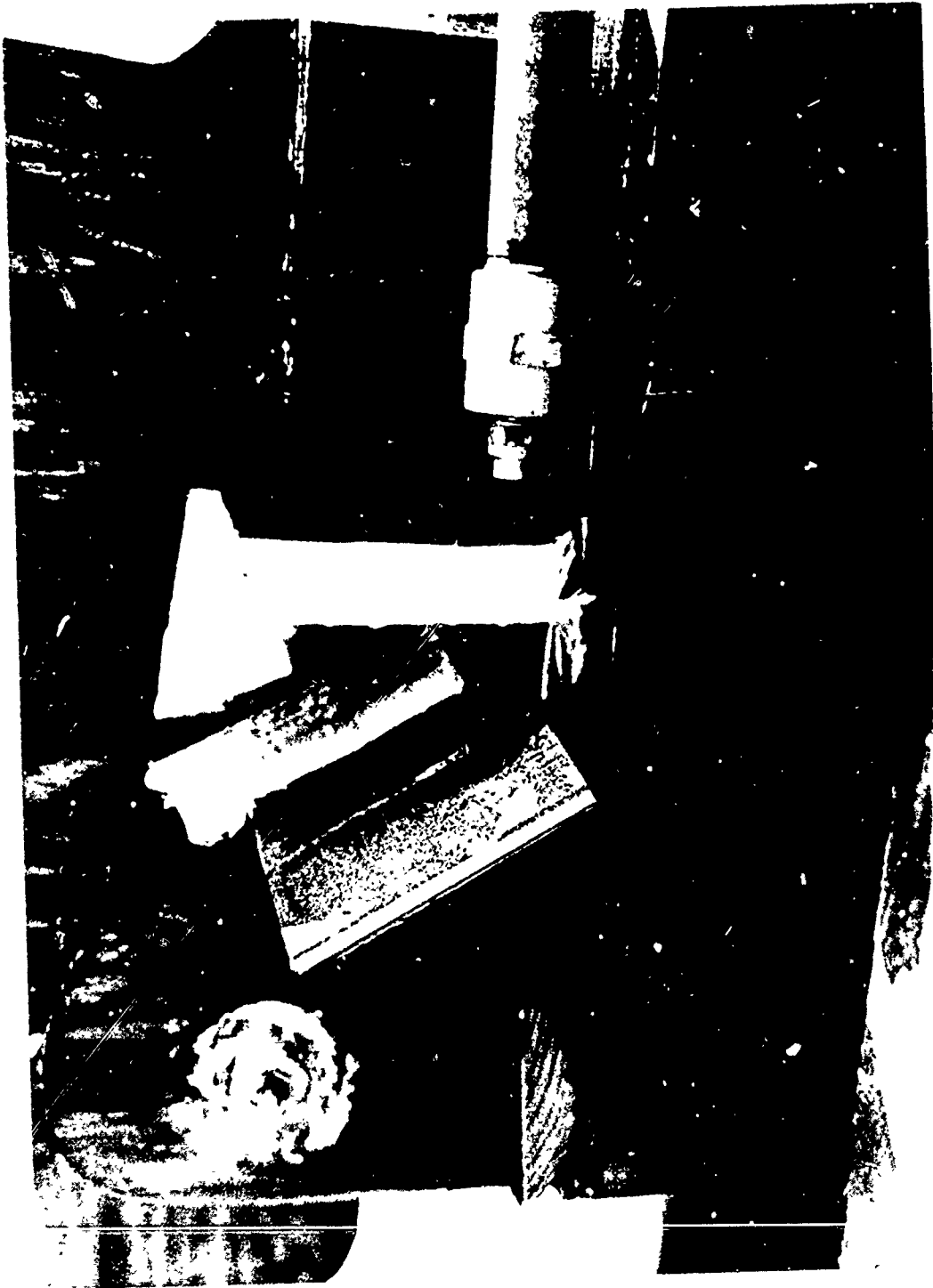
FIGURE 4. CLOSEUP OF WATER JET NOZZLE SHOWING THE TARGET, A SECTION OF MINUTEMAN III CASE WITH EXTERNAL INSULATION WEDGED IN THE HOLDER
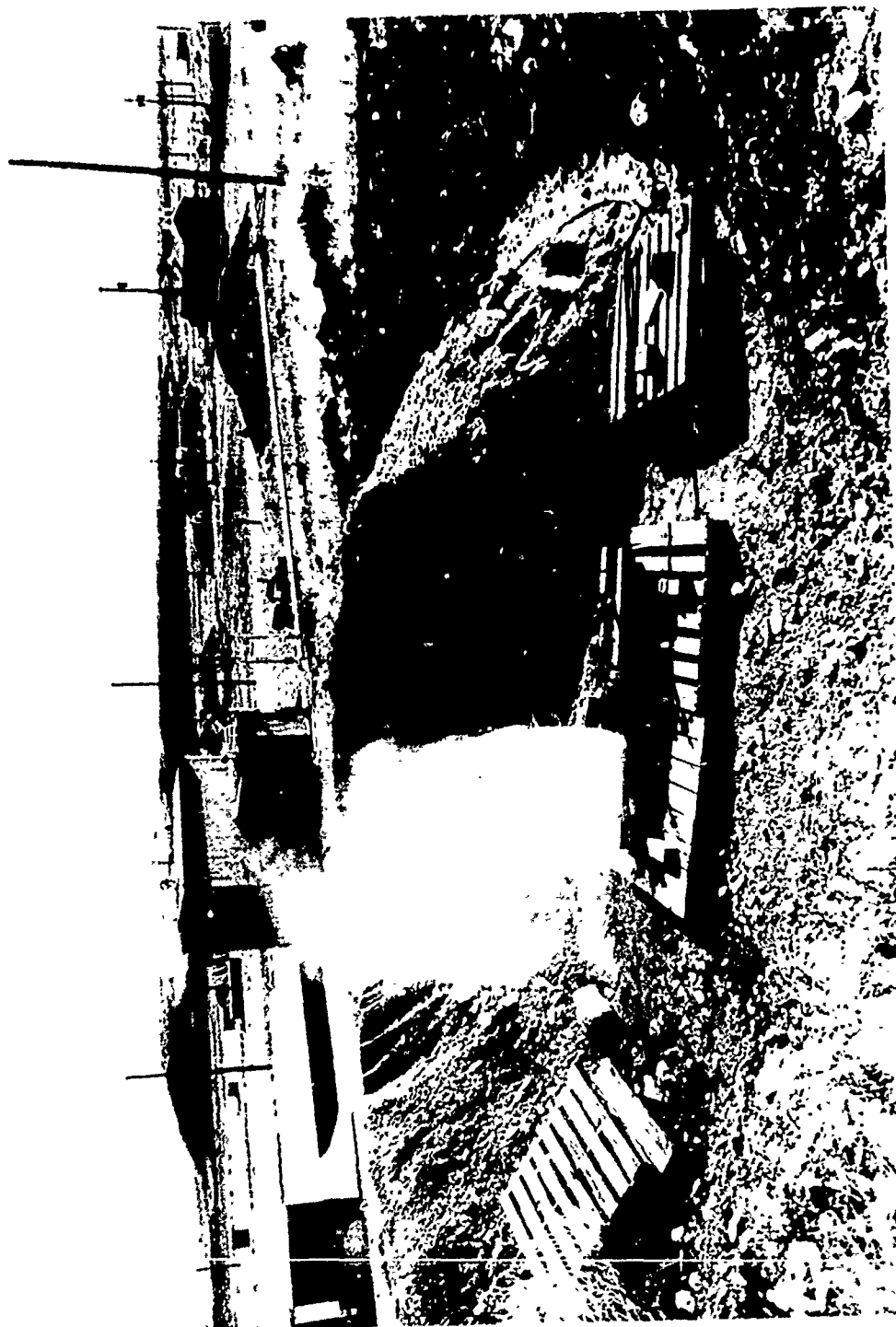
FIGURE 5. WATER IMPACT TEST SHOWING WATER IMPACTING A TARGET – SIDE VIEW

FIGURE 6. WATER IMPACT TEST. TEST SHOWING WATER IMPACTING A CASE SECTION AT
45° ANGLE. REAR VIEW

the pump is shown in the background. The test pit where the test fixture was located is shown in the far background between the bunker and the pump. Figure 2 shows the water blaster with the steel-braided, high-pressure hose attached to the pump outlet, just below the pressure gauge. Figure 3 shows the test fixture with water flowing. The target, either propellant or the insulation/case sample, was held in the wood clamp on the left of the fixture. When air was supplied to the cylinder via the vertical oil reservior on the right of the fixture, the lance was rotated until it impacted the target.

Figure 4 shows a closeup view of the nozzle and wood clamp with a sample of case with insulation in place. The abrasion of the insulation is clearly visible. Figures 5 and 6 show views of the fixture while water is impacting a target. The blocks on the pallet in the foreground of Figure 6 were used to hold the case/insulation samples at different impingement angles.

Blocks of propellant, obtained from casting propellant in ½-gallon ice cream cartons, were used for the impact and cutting tests. Initially a test duration of one minute was selected. The sweep time, time for the lance to swing from its starting position until it came to its stop point on the propellant, was determined by observations made during dry runs. Later, more accurate times were obtained by observing the water plume from the bunker entrance. The dwell time was measured from the time the sweep of the lance stopped until the power was cut on the pump motor reducing the pump pressure to its idle speed.

Figures 6 through 12 show selected views of the propellant after measuring the depth of the cuts. The propellant was cut along the path of the sweep cut. The average depth of the cut was measured. The dwell cut located in the center of the sample was also measured. Samples 146 through 151 were impact tests conducted at 10,000 psi water pressure and 190°F water temperature with a steel plate backing the sample. In Figure 12 the sample of CYH propellant had the propellant still bonded to the insulation and case section as it was cut from the motor. Although the insulation was not cut through during the sweep, both the insulation and the case were cut through during the dwell period.
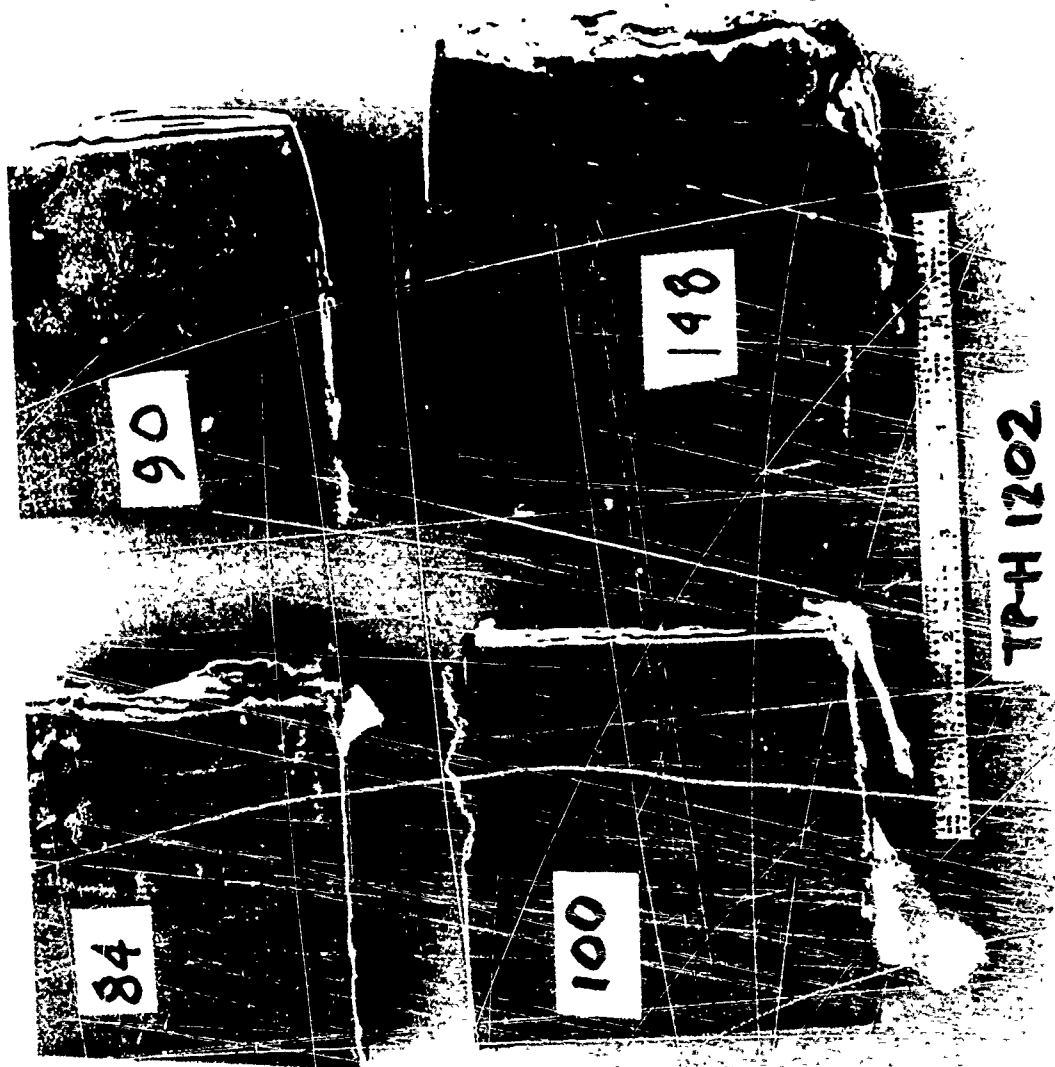
FIGURE 7. SAMPLES OF TP-H1202 PROPELLANT CUT BY WATER JET
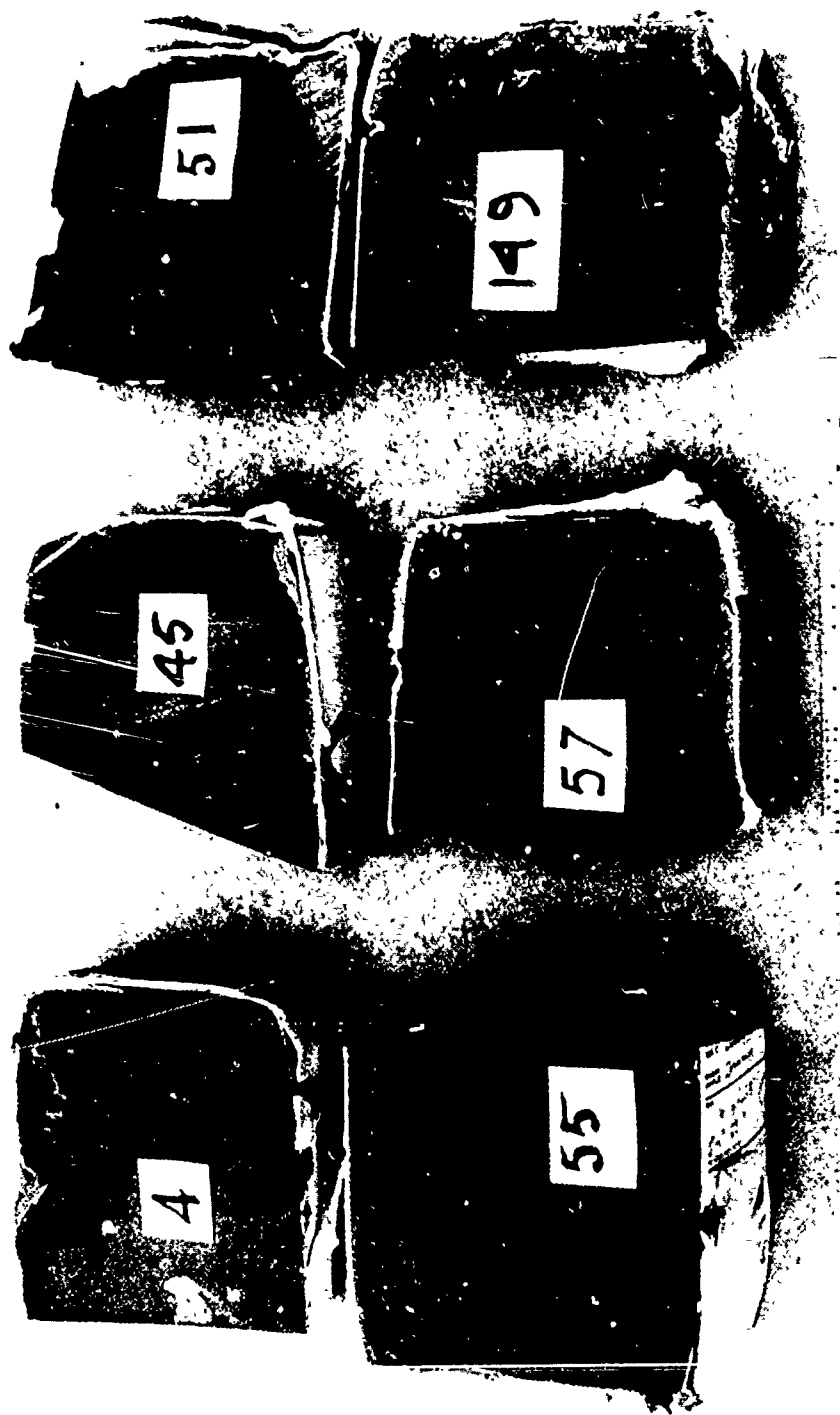
FIGURE 8. SAMPLES OF TP-H1207 PROPELLANT CUT BY WATER JET
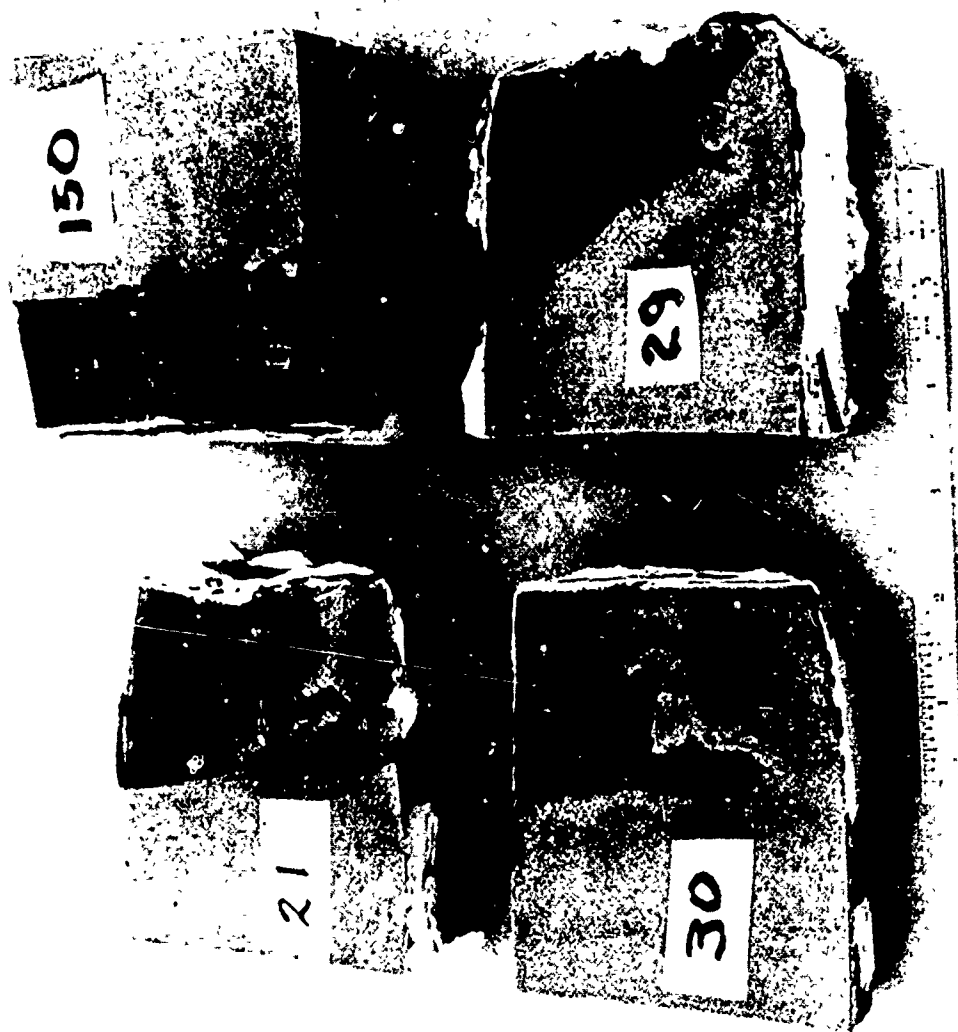
FIGURE 11. SAMPLES OF TP-H1035 PROPELLANT CUT BY WATER JET
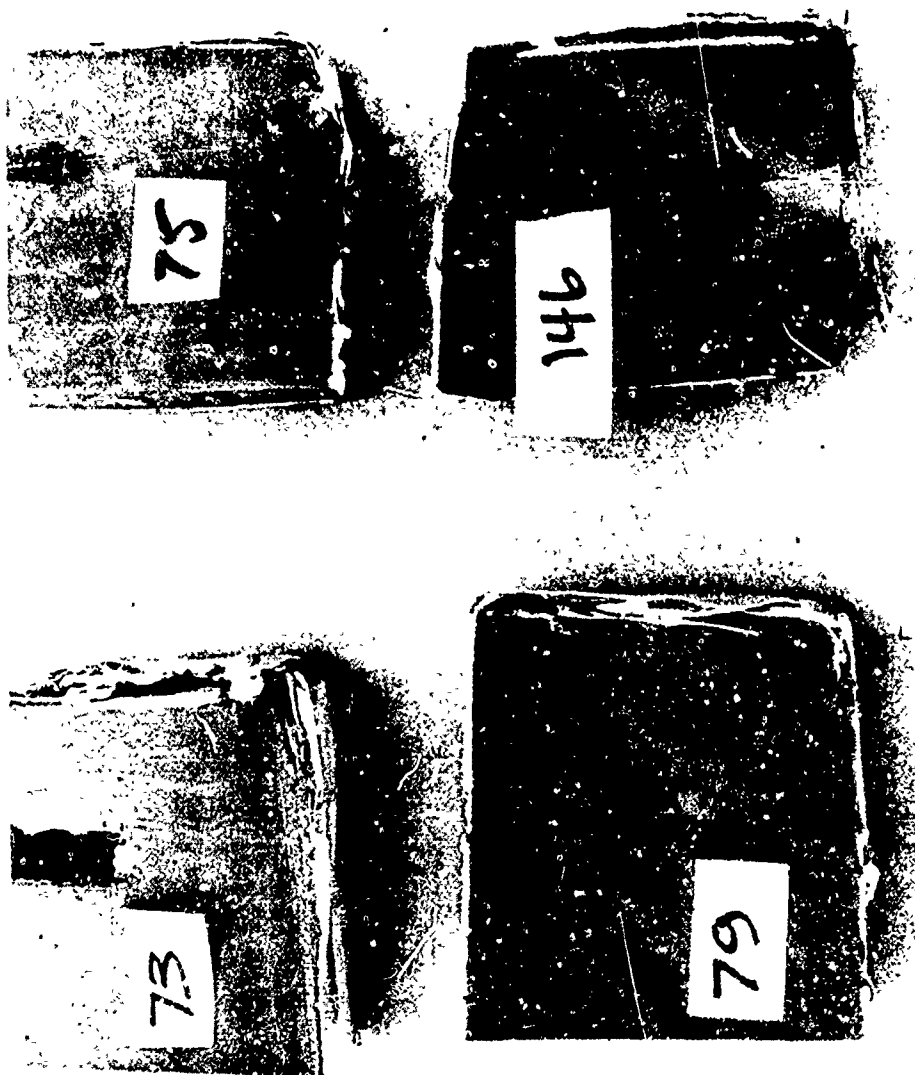
SAMPLES OF VRP PROPELLANT CUT BY WATER JET

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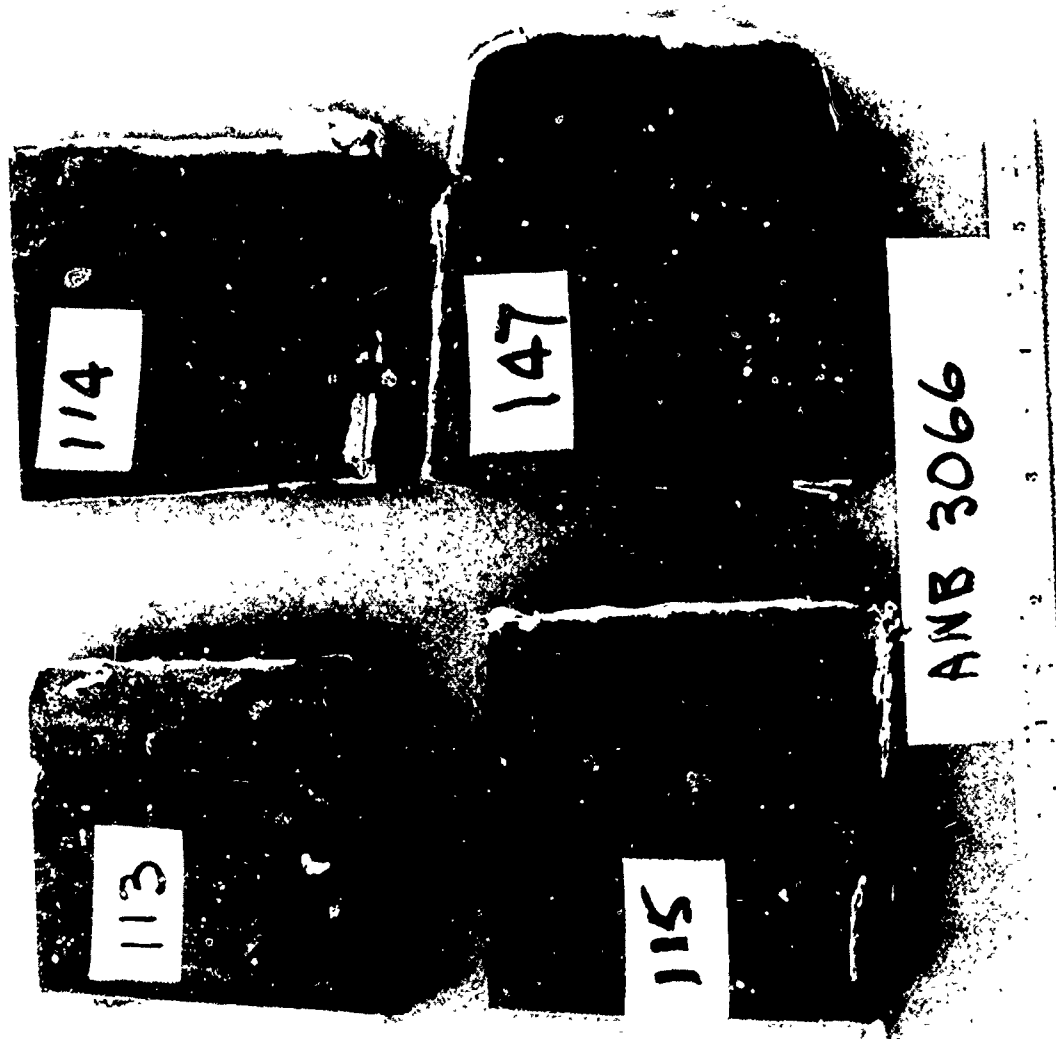
FIGURE 9. SAMPLES OF ANB-3066 CUT BY WATER JET
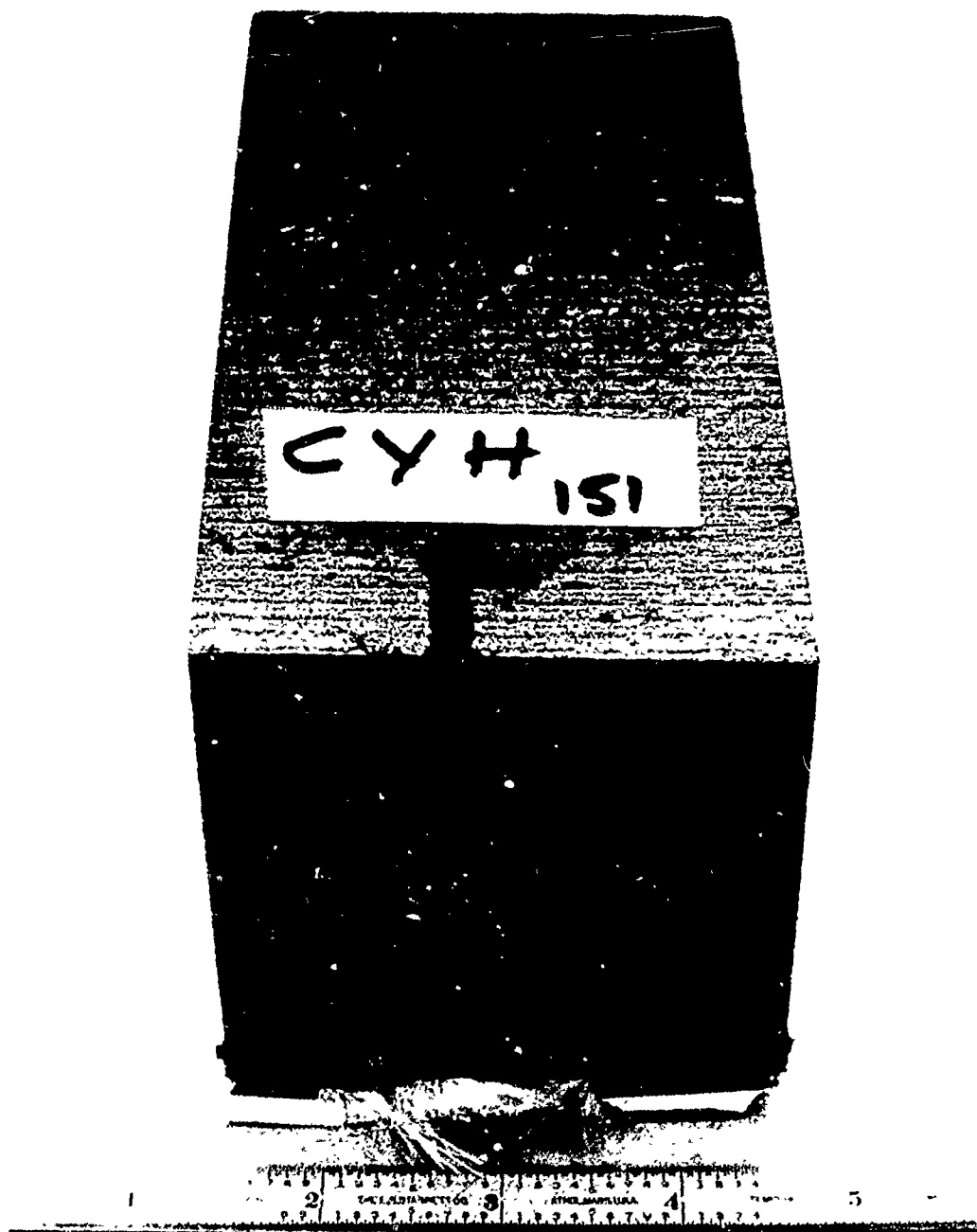
FIGURE 12.   SAMPLE   OF CYH PROPELLANT CUT BY WATER JET

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The data were analyzed by regression analysis to determine how the cutting rates varied as to type of propellant, water pressure and water temperature.

Figure 13 shows the normalized sweep cutting rate, the sweep cutting depth times the speed, versus the water pressure for TP-H1202 propellant. All data were regressed using a power function to fit the data. The resulting equation was:

$$\text{Cutting Rate} = 8 \ (10^{-6}) \ (\text{water pressure})^{1.8} \ \text{Square inches/second}$$

$$R^2 = 0.611$$
$$S_{yx} = 0.625$$

The data was then grouped by the water temperature and the data at each temperature was regressed. The results shown in Figure 13 indicate that there is little if any effect on the cutting rate due to temperature in this temperature range from 60°F to 190°F. The results are summarized in Table II.

Next a linear multiple regression program was used with temperature and pressure as the independent variables and the normalized sweep cutting rate as the dependent variable. The results, summarized in Table III are shown graphically in Figure 14. These results show that while using hot water had a definite effect of increasing the cutting effectiveness for ANB-3066 propellant, the effect was slightly less for TP-N1035 and VRP propellants. Increased temperature apparently had a slightly negative effect on the cutting effectiveness for TP-H1202 and TP-H1207 propellants. The low coefficient of correlation, R^2, for the TP-H1207 propellant is probably due to inaccuracy in determining the cutting time during the earlier impact tests.

The data were regressed using a linear curve fit for the normalized sweep cutting rate versus the water pressure. These results, plotted in Figure 15 show that that the cutting effectiveness or rate increases in the order of ANB-3066 > TP-H1035 > VRP > TP-H1202 > TP-H1207. Comparison of this order with the Shore A hardness of the propellant indicates this may be an inverse
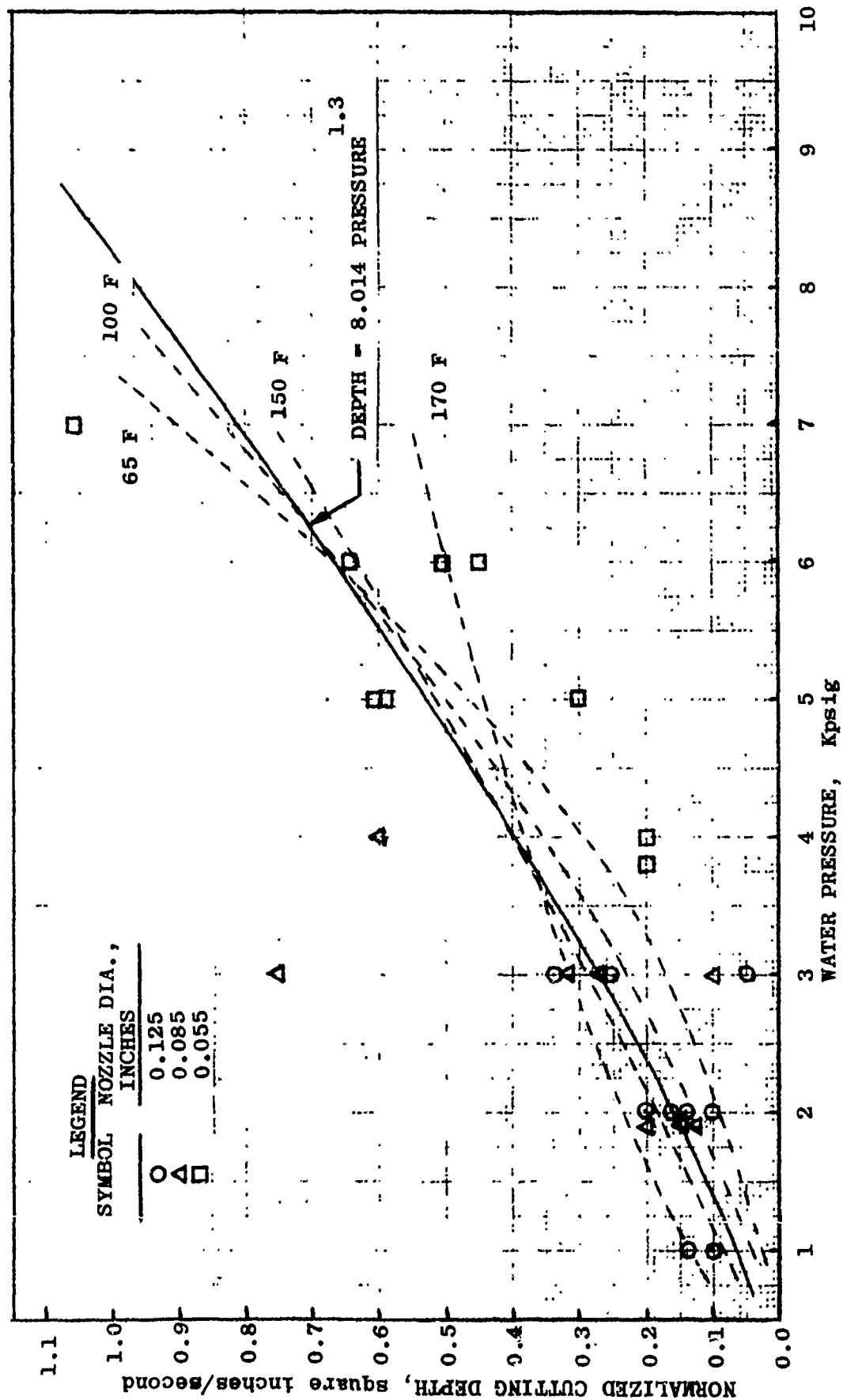
FIGURE 13. THE NORMALIZED SWEEP CUTTING RATE OF TP-H1202 PROPELLANT AS A FUNCTION OF THE WATER PRESSURE. (Assuming a power curve fit)

TABLE II. SUMMARY OF REGRESSION ANALYSIS* OF THE SWEEP CUTTING RATE FOR
TP-H1202 PROPELLANT AT VARIOUS TEMPERATURES

Temperature	A	B	R^2	δ	N
65	$2.87 (10^{-8})$	1.949	0.617	0.679	8
100	$1.08 (10^{-6})$	1.530	0.688	0.599	7
150	$4.40 (10^{-5})$	1.100	0.969	0.165	'
170	$1.75 (10^{-2})$	0.303	0.7122	0.189	3
Overall	$8.01 (10^{-6})$	0.300	0.6107	0.6254	24

* A power fit, $y = Ax^B$ was assumed.

TABLE III. SUMMARIZED RESULTS OF LINEAR MULTIPLE REGRESSION ANALYSIS OF
CUTTING RATE DATA FOR VARIOUS PROPELLANTS ($Z = A + Bx + Cy$)

Class	Propellant	A	B	C	R^2	N
1.1	VRP	-2.19	$2.85 (10^{-4})$	$3.39 (10^{-3})$	0.91	17
1.1	TP-N1035	-2.81	$2.57 (10^{-4})$	$4.86 (10^{-3})$	0.82	19
1.3	TP-H1202	-0.42	$2.49 (10^{-4})$	$-4.46 (10^{-5})$	0.68	27
1.3	TP-H1207	0.05	$1.50 (10^{-4})$	$-2.50 (10^{-4})$	0.33	36
1.3	ANB-3066	-3.50	$2.85 (10^{-4})$	$1.00 (10^{-2})$	0.96	10

TABLE IV. SUMMARY OF RESULTS OF LINEAR REGRESSION OF THE NORMALIZED SWEEP
CUTTING RATE (Y) VERSUS THE WATER PRESSURE (X)

($Y = A + BX$)

Class	Propellant	A	B	R^2	δ
1.1	VRP	-0.079	0.0003	0.656	0.722
1.1	TP-H1035	0.032	0.0003	0.717	0.652
1.3	TP-N1202	-0.054	0.0002	0.395	0.640
1.3	TP-N1207	0.158	0.0002	0.183	0.904
1.3	ANB-3066	-0.222	0.0003	0.888	0.342
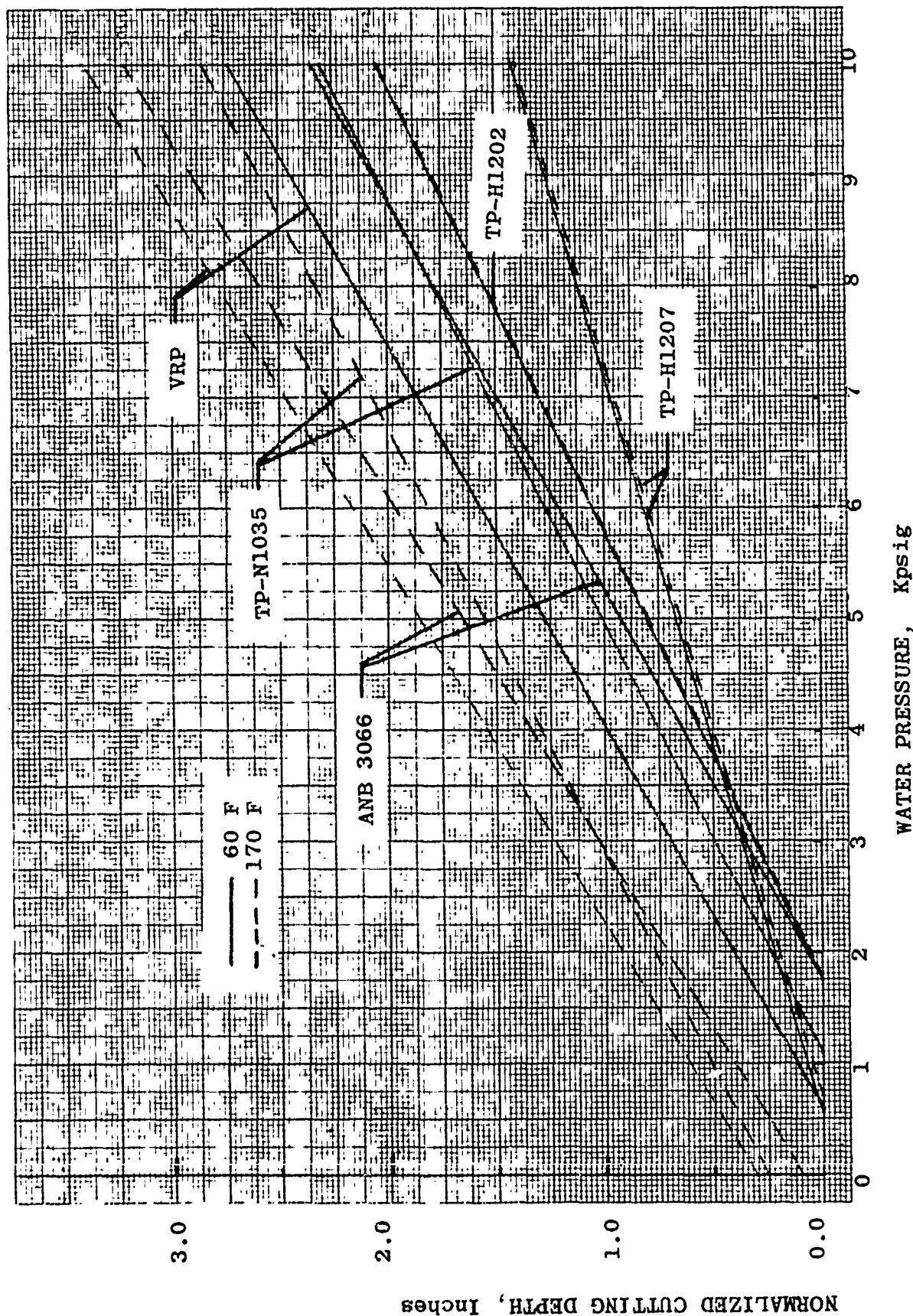
FIGURE 14. RESULTS OF LINEAR MULTIPLE REGRESSION OF SWEEP CUTTING RATES USING THE WATER TEMPERATURE AND PRESSURE AS THE INDEPENDENT VARIABLES. (Normalized cutting depth is the calculated cutting depth at a sweep rate of 1.0 inch per second.)
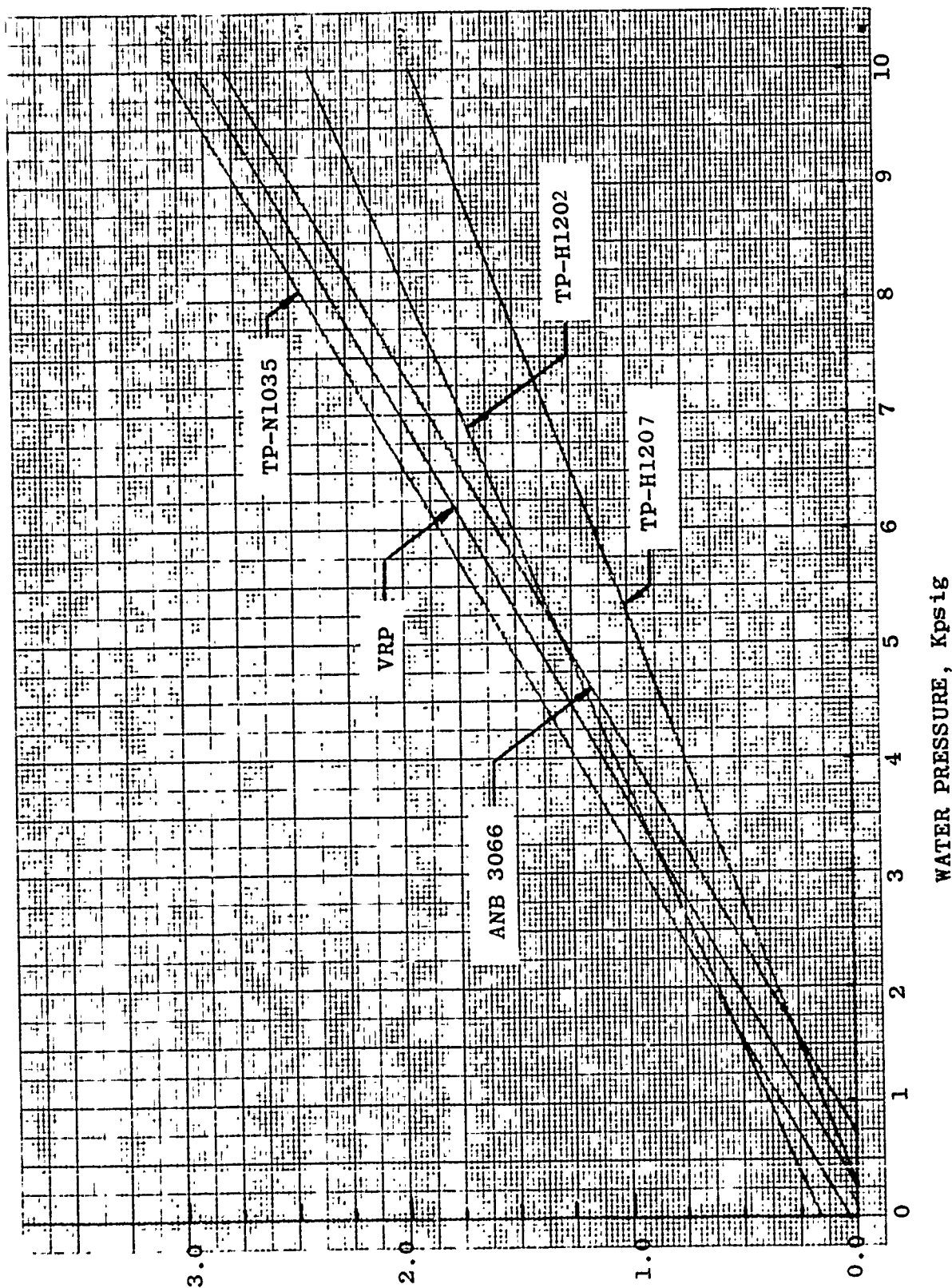
FIGURE 15. RESULTS OF LINEAR REGRESSION OF THE SWEEP CUTTING RATE AS A FUNCTION OF THE WATER PRESSURE. (Normalized cutting depth is the calculated cutting depth at a sweep rate of 1.0 inch per minute)

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function; i.e., cutting depth appears to be inversely proportional to the
Shore A hardness, except the order of TP-H1207 and TP-H1202 would need to
be reversed. The low value of R^2, the correlation coefficient, for the cutting
depth data of the TP-H1207 propellant indicates that there may be sufficient
error in the data for the suggested correlation to exist.

The cutting effectiveness of the nozzle was compared by plotting the lowest
pressure and temperature at which the propellant was cut through. The graph,
shown in Figure 16, ignores the slight variations in the sweep rate but does
give an indication of the dependence of the cutting rate on the nozzle size.
These data show that as the nozzle diameter increases, the cutting rate
increases. These data indicate that the general trend is that as the
temperature is increased, the cutting rate is increased.

Economically, the most efficient nozzle may be the smaller nozzle. As the
water flow rates increase with increased pressure and/or nozzle diameter,
consideration of the costs: 1) of water, 2) of removal of water from the
propellant waste, and 3) of heating the water becomes important. Table V
lists the water flow rates calculated for the range of pressures used during
the impact tests. The exit velocity was calculated based upon the cross-
sectional area of the nozzle.

In summary, hydromining appears to be a viable method of propellant removal
for both Class 1.1 and 1.3 propellants. A potential does exist for damage
to the case, particularly for Kevlar cases; however, shielding can be designed
to give adequate protection from the water. Damage to the insulation
can be minimized by utilization of hot water and low pressures for removal
of the propellant near the insulation. The basic cause for damage to the
insulation would result from stopping the travel and impacting the jet at
one location for too long a period of time. This could be avoided by having
an interlocking system which shuts off the water immediately when the
travel is stopped.
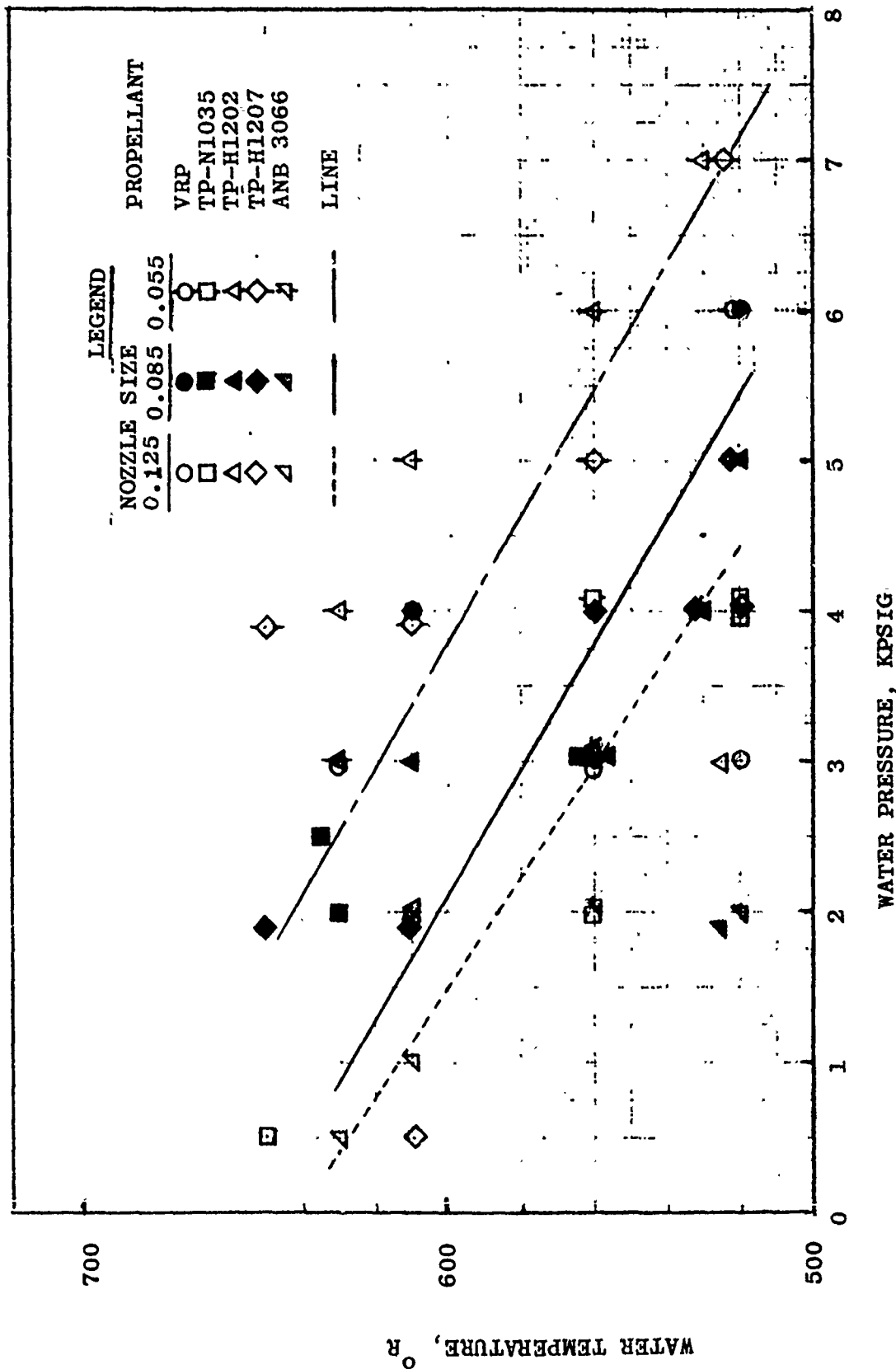
FIGURE 16. THE CUT-THROUGH POINT; i.e., THE TEMPERATURE AND PRESSURE AT WHICH THE WATER JET CUT THROUGH THE 4-INCH THICK PROPELLANT, AS A FUNCTION OF , THE NOZZLE SIZE.

TABLE V. WATER FLOW RATES FOR THE AMERICAN AERO PUMP. FLOW RATE (GPM) = 0.00976 TIMES PUMP SPEED (RPM). PUMP PRESSURES CORRESPOND TO PUMP SPEED AS OBSERVED DURING TESTING.

Nozzle Size	Pump RPM	Flow GPM	Velocity Ft/Sec	Pressure psi
.125	500	4.88	127.5	
	600	5.86	152.7	
	700	6.83	178.5	500
	800	7.81	204.2	750
	900	8.78	229.5	900
	1000	9.76	255.2	1000
	1500	14.64	382.7	2000
	2000	19.52	510.3	4000
	2200	21.47	561.3	10000
.085	500	4.88	275.9	1800
	600	5.86	331.3	
	700	6.83	386.1	3000
	800	7.81	441.5	
	900	8.78	496.4	5000
	1000	9.76	551.8	6000
	1500	14.64	827.8	10000
.055	500	4.88	659.0	3800
	600	5.86	791.3	5000
	700	6.83	922.3	7000
	800	7.81	1054.6	9000
	900	8.78	1185.6	10000

6.1.2   Machining

The objective of the milling tests were to determine the optimum milling
conditions for different propellants and attempt to correlate milling
costs with the characteristics of the propellant. The primary concern was
to determine if class 1.1 propellants could be milled at sufficiently high
rates as to be economical.

The method of investigation to be used was to experimentally mill various
propellants at blade tip speeds from 2 to 3 feet/second at cutting depths
of 1/8 to 1/2 inch and measure the cutting force required and the blade
temperature at the different propellant removal rates. Using heat transfer
and thermodynamic data for the respective propellants, the heat flux could
be calculated and compared with the ignition time as determined from arc
image furnace tests.

Because an extensive amount of data was found in literature and in unpublished
Thiokol reports, the tests were somewhat modified to attempt to improve
milling speeds above what is currently practiced.

It was concluded from the results of this effort that any of the listed pro-
pellants (VRP, TP-N1035, CYH, TP-H1202, TP-H1207, ANB-3066) could be safely
dry-milled at cutter tip speeds up to 28 feet/second and feed velocities up
to 15 inches/minute with no measurable temperature rise in the propellant.
The principal hazards which highly recommend the wet machining operation over
the dry machining operation are potential ignition due to tool breakage or
foreign objects in the propellant. The cutting rates achieved with a new
cutter used in these tests appear to be much higher than is currently
achieved using tooling developed for laboratory sample milling operations
and C4 cutback operations. New cutting rate predictions will be determined
for use in the cost model developed in Phase II based upon these cutting
rates and those determined by hazards analysis.

Teletemp indicator dots were previously used at Thiokol to attempt to measure
the blade temperature during machining to remove inert C4 propellant from
motor TD-0014. A sketch of the machining blade is shown in Figure 17.
Cutting parameters were: Rotational speed = 50 rpm; cutting depth - 6-7 inches;
feed rate = 2.4 inches/minute. The initial propellant and blade temperature
was 70°F. Two tests were conducted and no dots discolored indicating the
temperature increase was less than 100°F. Subsequently, 100 and 110°F
teletemp dots were used during initial machining cuts of the TD-0014 motor.
The initial temperature was recorded as 81°F. During six separate cuts, a slight
discoloration was observed on single 100°F dots during three of the tests.
No discoloration was observed on 110°F dots. This indicated a maximum tempera-
ture increase of the cutting tool of 19-29°F.

An attempt was made to use a Barnes infrared thermometer to measure the
propellant surface temperature during machining. A special 5-inch diameter
butterfly cutting tool was designed to allow the thermometer (in an explosion
proof box) to be placed near the propellant being machined as shown in Figure 18.

The temperature thermometer was calibrated at 90 and 200°F points and assumed
linear between 75 and 250°F. During the cutting process, the thermometer
remained at 75°F. To verify that the thermometer was capable of measuring
under these conditions, a block of propellant was heated to 160°F then placed
in the mill vise. The reading of 120°F indicated that the thermometer reads
low for this spectral color of propellant (non-black body). It was concluded
that the thermometer could not read the blade or propellant temperature
accurately.

The cutter was then used for a series of machining tests. At the end of each
cutting test, the propellant surface and the blade were felt by hand and no
noticeable temperature increase was detected. The range of parameters
tested were: cutter rotational speeds (18 to 1300 rpm), feed velocities
(0.5 to 15 inches/minute), cutting depths (0.2 to 0.5 inches). A significant
improvement of this cutter over those currently being used for machining
propellant blocks was that the chips and/or ribbons of propellant pass
through the hole in the cutter and are thrown into the milling tray and not
accumulated on the propellant surface (See Figure 19). It was concluded

DOT LOCATIONS

5 INCHES

100 & 120 F DOTS ON ONE SIDE

130 & 150 F DOTS ON OPPOSITE
SIDE
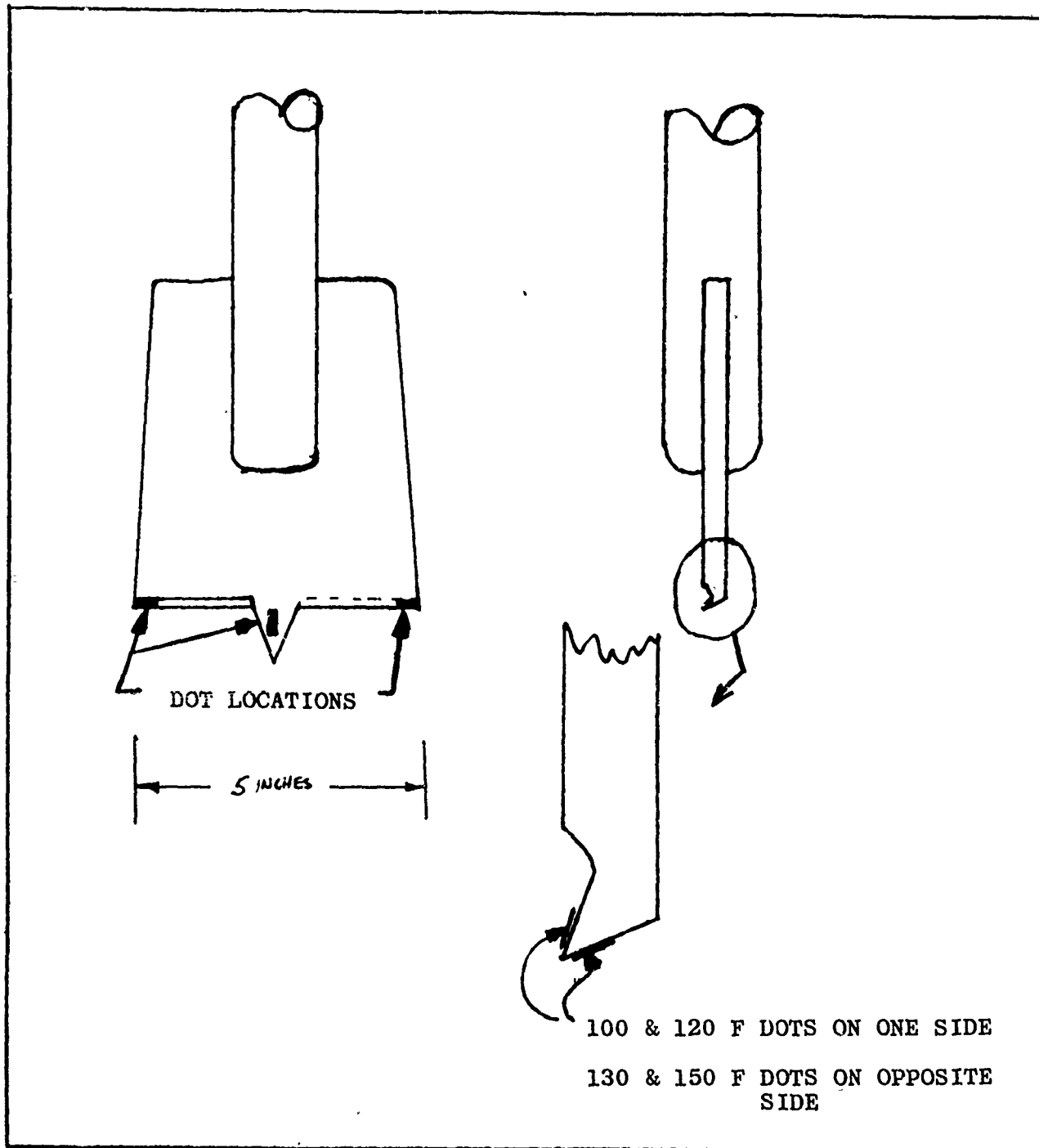
FIGURE 17. SKETCH OF SPADE DRILL BLADE USED TO MACHINE C-4
PROPELLANT FROM MOTOR FD-0014 SHOWING LOCATION OF
THE TELETEMP INDICATOR DOTS.

FIGURE 18. SET-UP OF MACHINING TEST SHOWING LOCATION OF THE BARNES INFRARED THERMOMETER RELATIVE TO THE BUTTERFLY CUTTER AND THE PROPELLANT.

FIGURE 19. PROPELLANT MILLED BY SPECIAL BUTTERFLY CUTTER SHOWING RIBBONS OF PROPELLANT REMOVED AND THE SMOOTH PROPELLANT SURFACE.

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that the cutter dissipates the heat rapidly and the heat rise in the pro-
pellant due to compression and friction was too slight to observe.

A hazard analysis of the cutter was made (Appendix A) with the method of
Hikida.[2] The results of the analysis, given in Appendix A, were admittedly
conservative but do demonstrate the type of analysis that is available and
that can be performed in the design of any machining operation that would
be performed during case salvage operations. An additional precaution that
should be taken in high speed machining of NG-type propellants is to
determine if the desired speed predicts temperatures which would produce
NG decomposition. An experimental monitoring method would be to use a LIRA
analyzer to look for NG decomposition products, e.g., nitrogen oxides, in
the chip removal duct or near the propellant surface. Speeds that produced
detectable decomposition could thus be avoided.

The arc-image furnace has been used extensively as a laboratory tool for
characterizing the ignitability of solid propellants. Since the ignition
source is radiant energy, a fraction of the radiant energy is reflected and
the delivered heat flux must be presumed greater than that required to ignite
the propellant. A fraction of the radiant energy is absorbed into the interior
of the propellant since it is not opaque to radiation. It is therefore
argued that ignitability data from arc-image furnace tests can be misleading
and is not representative of heat transfer to propellant where convective
or conductive modes of transfer are predominant. A paper was found describing
a correlation between solid propellant arc-image data with the propellant
burn rate. This predictive capability plus the data already available from
various souces was judged to be sufficient to fulfill the needs of this
program. Measures to obtain specific arc-image data was terminated.

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[2] Hercules Incorporated memorandum MISC/6140-3033 (Hazards), "Analysis of
Heat Generation from Dry Machining of Solid Propellant," E. T. Hikida.
August 1972.

For composite propellants, the time to ignition as a function of the radiant heat flux can be obtained from the burn rate of the propellant according to the correlation developed by Derr and Fleming[3] shown in Figure 20. Data for VSB and VRA propellant, close analogs to VRP propellant are given in Figure 21. Ignition and burn rate data for VTG-5A propellant are given in Figures 22 through 24 These examples are typical of what is available in literature. These data are acceptable for the use proposed in this study. Development of a comparable correlation to Derr and Fleming's for crosslinked, double-base propellants, if it does not yet exist, is considered to be beyond the scope of this study.

A compilation of the safety data and mechanical properties data for the six propellants is given in Tables VI and VII, respectively.

In summary, it was concluded that the bulk of the propellant can be safely removed by either wet or dry machining. Dry machining would eliminate any potential damage water may do to the case. Wet machining requires taking precautions to eliminate contact of water with the case. This is successfully done during cutback operations on C4 motors where VRP propellant is removed from the forward end by wet machining. Removal of the last inch or two of propellant near the insulation would increase the potential of damage to the insulation. Low pressure hydromining remains the best method for removal of the residual propellant.

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3) Derr, R. L. and Fleming, R. W., "A Correlation of Solid Propellant Arc-Image Ignition Data", Lockheed Propulsion Company, Redlands, CA.
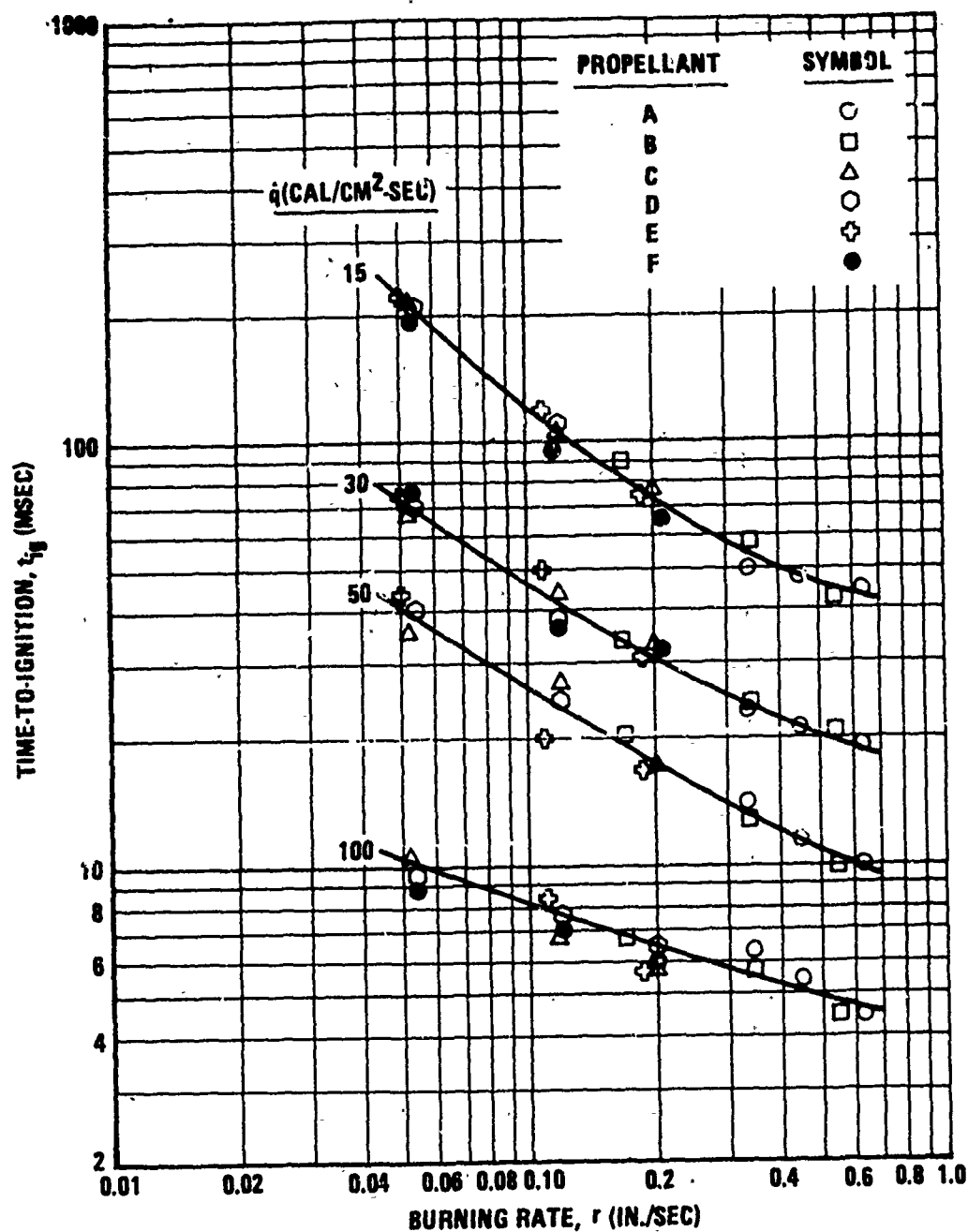
FIGURE 20. CORRELATION OF TIME-TO-IGNITION WITH BURNING RATE.
(ZrC Coated Samples)
Source: R.L.Derr and R.W.FLeming, "A Correlation
of Solid Propellant Arc-Image Ignition Data"
Lockheed Propulsion Co., Redlands Ca.
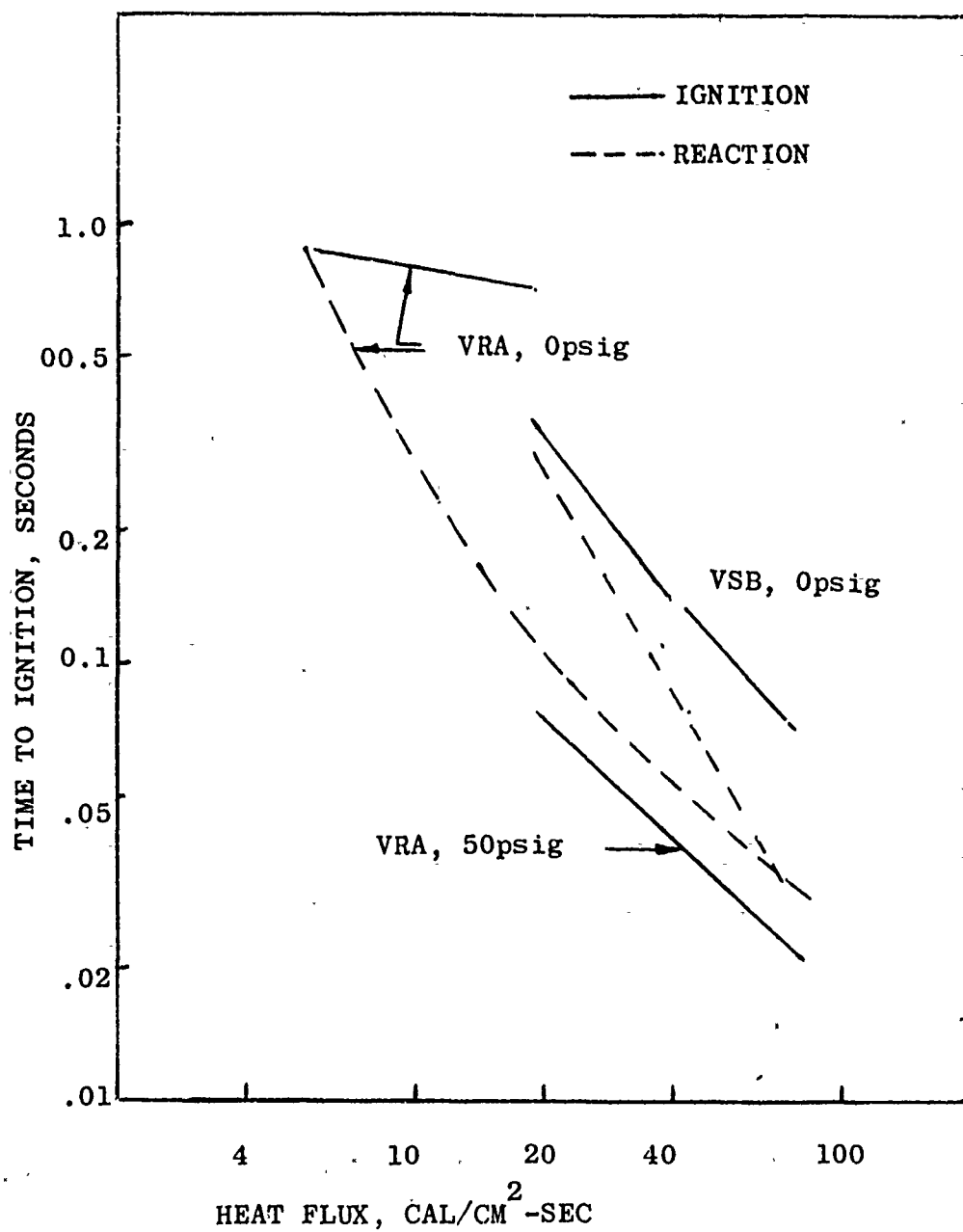
FIGURE 21. ARC-IMAGE IGNITION RESULTS. COMPARISON OF VSB
AND VRA PROPELLANTS. (Source: K.B.Isom, Hercules
Inc., Bacchus, Utah)
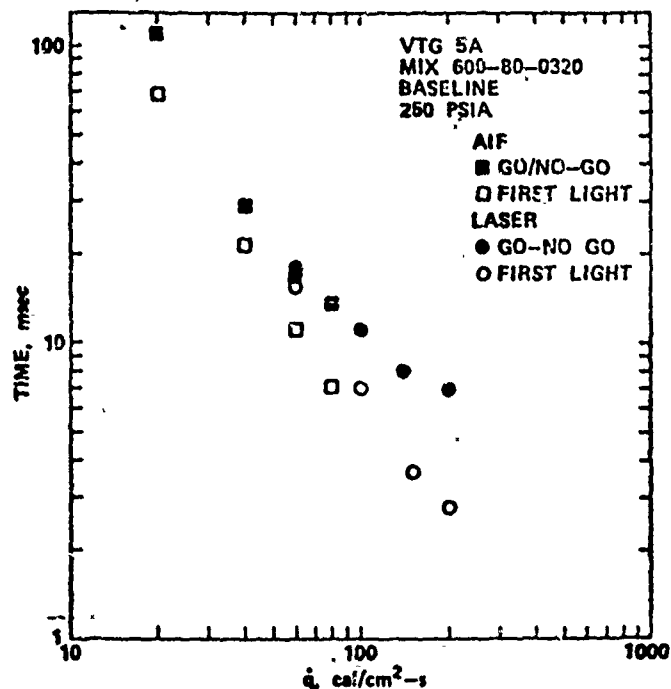
FIGURE 22. BASELINE LASER IGNITABILITY DATA FOR 250 PSIA FOR
VTG-5A PROPELLANT.(Source: A.I.Atwood et.al., "The
Effect of Aging on Ignition of Trident VTG-5A
Propellant",NWC, China Lake, Ca.)
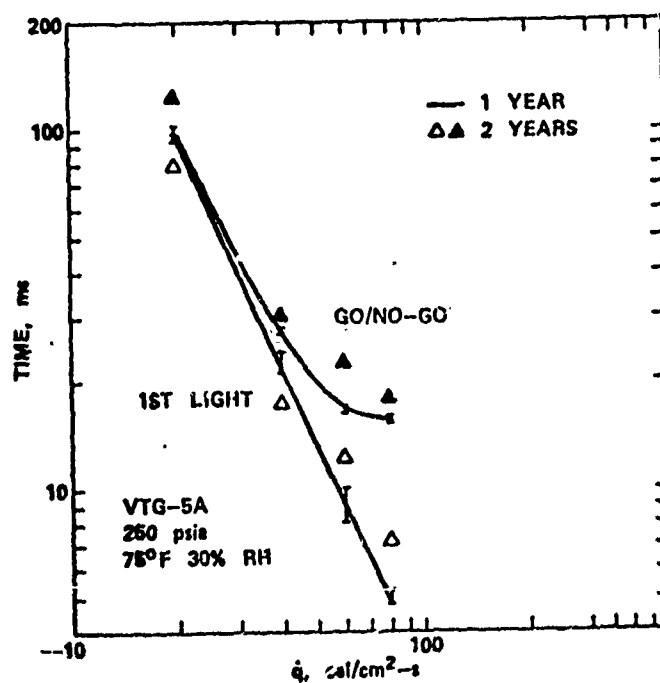
FIGURE 23. IGNITABILITY OF AGED VTG-5A PROPELLANT. AGED AT 75 F
AND 30% RELATIVE HUMIDITY. (Source: A.I. Atwood, et al)
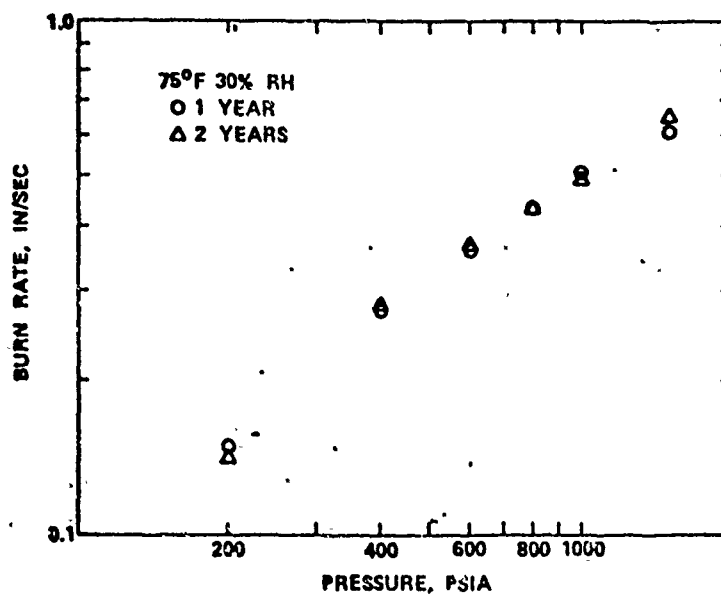
FIGURE 24. BURN RATE OF AGED VTG-5A PROPELLANT. AGED AT 75 F
AND 30% RELATIVE HUMIDITY.
Source:A.I.Atwood, et.al., "The Effect of Aging on
the Ignition of Trident VTG-5A Propellant", NWC,
China Lake,Ca.

TABLE VI. MECHANICAL PROPERTIES DATA FOR PROPELLANTS USED IN CASE SALVAGE EVALUATION

Propellant	Density, lb/in^3	Stress (σ), psi	Strain (ε), %	Modulus (E), psi	Shore A	Penetrometer, mm
VRP	0.068	110	195	370	35	27
TP-N1035	0.066	70	175	675	46	23
TP-H1207	0.065	145	51	600	50-60	25
TP-H1202	0.0656	150	32	800	67	16
ANB-3066	0.0638	91	37	396	--	17
CYH	0.0640	170	50	690		6

TABLE VII. SAFETY DATA FOR PROPELLANTS USED IN CASE SALVAGE EVALUATION

Propellant	Impact, in.*	Friction, lb.	Autoignition & Fisher-John	Burn Rate
VRP	12.3 (1.5)	46.4 (2.7)	26 min. @ 300°F No ignition @ 225°F 430-441°F @ 27 °F/min.	OPC R_B 1000 = .438 Slope = 0.48
TP-N1035	8.41-9.50 (.79 to 1.23)	>64	.59-.63 hrs @ 300°F >24 @ 225°F	TU628 R_B 1000 = 0.430 Slope = 0.55
TP-H1207	11.9 to 58.2 (2.69 to 0.91)	58.2 to 64 (0.91 to 4.14)	>24 @ 300°F	TU628 @ 1565 = 0.675
TP-H1202	19.4 to 20.1 (2.1 to 1.57)	58.2 to >64 (4.0 to 2.6)	>24 @ 300°F	TU628 @ 1000 = 0.332 Slope = 0.32
ANB-3066	12	55	>24	

*Standard deviation is included in parenthesis.

6.1.3   Burn Out

The simplest, most direct method of removing the propellant is to burn it
out. Burnout under controlled conditions, similar to a static test firing,
would remove propellant, flap, liner and part of the insulation and would
further eliminate the cost of propellant waste disposal. The currently
unanswered question about this method is the assessment of the risk of
damage to the case due to possible localized heating which could occur.
To answer this question, a computerized heat transfer study was initiated.

Two motors, first stage MX and third stage Minuteman, have been analyzed
to determine whether reduced pressure burning is feasible from a thermal
standpoint.

For relatively high pressure motors such as first stage MX, reduced pressure
burning is not feasible from two aspects. First, the case/insulation interface
temperatures reach unacceptable levels (895F) leading to case degradation. Second,
slag accumulation burns completely through the insulation and through several
windings of the Kevlar case leaving it unsuitable for reuse.

For low-pressure motors such as third stage Minuteman, reduced pressure
burning is a high risk method of case salvage. Case/insulation interface
temperatures are marginally acceptable. A CO_2 quench rather than water would
be necessary. Slag accumulation varies a great deal in this type of motor
but would always be a potential cause of case degradation.

The thermal analysis was performed using CMA, an Aerotherm one-dimensional
heat ransfer computer program which calculates the temperatur ;radient
through multiple material layers and accounts for decompositio. and ablation.
This program has been used successfully on many motor designs to evaluate
insulation performance. Several locations in the motors were analyzed at
reduced pressure levels to evaluate the case/insulation interface
temperature. The thermochemistry input for the different propellants and
pressure levels was calculated using the Aerotherm Equilibrium Chemistry
(ACE) computer program. The combustion gas composition and properties were
calculated using the NASA Lewis Chemical Equilibrium computer program at
each pressure level.

Static test data from the MX and Minuteman programs was used to determine
the boundary conditions at various locations in the motors by varying the
heat transfer coefficient to match the measured insulation loss rates. Once
the heat transfer coefficients were known at the standard operating pressure
level, they were then adjusted for the reduced pressure level by:

$$h_r = (\frac{P_r}{P})^{.8} h$$

Where: h_r = heat transfer coefficient at reduced pressure

P_r = reduced pressure value

P = normal operating pressure

h = heat transfer coefficient at normal pressure level

Other boundary conditions including radiant heat flux, recovery enthalpy
and thermochemistry were calculated using the above mentioned programs.

The propellant burn rate equations were known from characterization studies
and the normal burnback pattern was modified for the new burn rate corresponding
to the reduced pressure level. This gave the time the insulation was exposed
to chamber conditions at the various locations.

The case/insulation interface temperature results are shown in Table VIII for MX. The hottest location is in the aft cylindrical area of the motor. To determine the worst case, quench effects were not included. However, because the temperature reaches its peak soon after burnout, a quench would not be helpful in reducing the temperature at the worst location. (Location 5, center area of the cylinder.)

In three static tests of the first stage MX, the slag accumulation varied from 76 to 213 pounds. The slag extended the length of the cylindrical section and had a width of up to two feet. Despite a high flow rate CO_2 quench, the slag burned through the insulation and through several windings of the Kevlar case. The large amount of slag virtually eliminates low pressure burning as a case reclamation technique for this motor.

Since the normal operating pressure of third stage Minuteman is relatively low, only one reduced pressure (200 psi) level was analyzed. The results are shown in Table IX. The presence of stress relief flaps which do not completely erode prevents accurate assessment of boundary conditions in the outboard areas of the forward and aft domes and thus the number of analyzed locations was decreased. However, the locations shown are believed to show the general effect of reduced pressure operation. Since a water quench does not provide uniform cooling, a CO_2 quency would be necessary to minimize pot-burn heat soak effects.

Slag accumulation varies greatly in the Minuteman motor ranging from .1 pound to over 10 pounds. The amount of slag cannot be accurately predicted but since the alumina particle size increases with decreasing pressure, the accumulation is expected to be greater at reduced pressure. The possibility of enough slag to damage the case would therfore be quite likely.

In summary, for motors which normally operate at high pressure, the large increase in exposure time will result in excessive case/insulation interface temperatures. Low pressure motors will not experience such a large exposure time increase and temperatures may remain acceptable. Slag accumulation will

TABLE VIII. First Stage MX Temperature Prediction

Location*	Pressure (psia)	Exposure Time (sec)	Peak Case/Insulation Interface Temperature (°F)	Time After Burnout (sec)
1	200	125	397	200
2	200	125	79	300
3	200	97	295	300
4	200	48.6	446	60
5	200	9.7	895	4
1	600	77	301	180
2	600	77	81	300
3	600	59.9	234	300
4	600	30	348	60
5	600	6	690	6
1	900	65	278	180
2	900	65	85	300
3	900	50	232	300
4	900	25.3	320	40
5	900	5.1	631	6

*Locations

1 = Forward polar boss

2 = Aft polar boss

3 = Aft Y joint

4 = Aft end of cylinder

5 = Center area of cylinder

TABLE IX.   Third Stage Minuteman Temperature Predictions

Location*	Pressure (psia)	Exposure Time (sec)	Peak Case/Insulation Interface Temperature (°F)	Time After Burnout (sec)
1	200	71.9	299	60
2	200	61.4	274	120
3	200	6.2	326	26
1	525**	54.9	297	120
2	525**	46.9	251	120
3	525**	4.7	290	24

*Locations

    1 = Forward polar boss

    2 = Aft polar boss

    3 = Aft end of cylinder

**Normal Operation Pressure

present a high risk of case damage unless taken into account in the initial insulation design. Such a redesign could provide acceptable low pressure performance but would carry a flight performance penalty due to added weight.

It was concluded that the normal window bomb tests would not verify the above results. In the window bomb tests, the propellant burns normal to the surface and no insulation is exposed until the instant of final burnout. This does not compare to the severe condition described above nor does it represent the extended duration of heat transfer that would occur during the quench period. Modification to the window bomb test was examined to obtain a more realistic test. This attempt was unsuccessful and development of a test method for this purpose was deemed to be beyond the scope of this task. As a secondary removal method, burning the residual propellant decreases the risk to the case but the economic advantage is lost. It is feasible to remove most of the propellant by machining and then burnout the last 1-2 inches next to the insulation. Agglomerate deposition would be greatly decreased but the cost of removal, for ignition devices and reusable nozzles, would be greater than removing the balance of the propellant by low-pressure hydromining; hence, this method has not been considered further. As a primary removal method, the risks of case damage are too great to be generally accepted. This does not preclude that an individual case might be developed where burn out could be effective.

6.1.4 Solvent Degradation of the Propellant

From an economic standpoint the utilization of solvents for propellant removal appears to be untenable. First, there is the relatively high cost of the solvents (unless water or steam is acceptable). Next, there are the higher equipment costs for handling the solvents, most of which are toxic or hazardous, and for their recovery for recycle. Finally, there is the increased potential of damage to the insulation and case. The reasons for conducting the solvent degradation tests are briefly:

1. Can the use of solvents desensitize the propellant to enable it to be removed more safely (particularly applicable to class 1.1 propellants).

2. Can degradation of the propellant during removal be integrated into the overall salvage operation to incorporate propellant ingredient recovery into the waste disposal process; thus, defraying the case recovery cost with benefits derived from ingredient recovery.

The format for solvent degradation tests was as follows:

1. Identify solvents which cause degradation of specific propellants.

2. Determine the hazards associated with solvents and with propellant/solvent mixtures.

3. Identify the effects of solvents on insulation.

4. Identify the effects of solvents upon the case.

5. Identify the effects that solvents may have on relining and subsequent reloading of the case.

6. Identify the effects that solvents may have on rebonding flaps and/or additional insulation to the remaining insulation.

Twenty-eight solvents were selected for testing the six propellants for degradation and/or desensitization. This does not preclude the possibility that another solvent may be better, but the solvents chosen were selected as likely candidates based upon references reviewed during the literature search in Phase I. These solvents are listed in Table X together with data useful in assessing potential hazards associated with their usage.

TABLE X. SOLVENTS USED IN SCREENING TESTS FOR PROPELLANT DEGRADATION

Solvent	Flash Point (°C)	Boiling Point (°C)	TSL*	Caution
Acetone	-18	56	AL31500	
Acetonitrile	6	82	AL77000	(Lachrymator)
Benzene	-11	80	CY14000	(Cancer Suspect Agent)
Carbon disulfide	-30	46	FF66500	(Stench)
Carbon tetrachloride	--	77	FG49000	(Cancer Suspect Agent)
Chloroform	--	61	FS91000	(Cancer Suspect Agent)
Cyclohexane	-20	80	GU63000	
Dimethylformamide	58	153	LQ21000	
Dimethylsulfoxide	95	189	PV62100	(Penetrates Skin)
p-Dioxane	12	101	JG82250	(Cancer Suspect Agent)
Ether	-45	34	KI57750	
Ethyl acetate	-4.4	77	AH54250	
Ethylene glycol	111	197	KW29750	
Hexane	-22	68	MN92750	
Methanol	12	64	PC14000	
Methylcyclohexane	-4	101	GV61250	
Methylene chloride	--	40	PA80500	
Methyl ethyl ketone	-7	80	EL64750	
1-methyl-2-pyrrolid in one	95	202		
1-propanol	25	97	UH82250	
2-propanol	12	82	NT80500	
Tetrachloroethylene	--	121	KX38500	(Irritant)
Tetrahydrofuran	-14	66	LU59500	
Tetramethylene sulfone	165	285	XN07000	
Toluene	4	110	XS52500	
Trichloroethylene	--	88	KX45500	(Cancer Suspect Agent)
2,2,4-Trimethylpentane	-12	150	SA33200	(Moderately toxic)
m-Xylene	29	139	ZE24500	

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*The TSL No. (Toxic Substance List Number) is included to emphasize that safety aspects are   important in the final selection of solvents for use in case reclamation. During the laboratory phase of this investigation, all known hazards associated with use of each solvent were   considered and a safe technique for its use was employed.

Screening evaluations · of the effects of twenty-eight solvents on six
different propellants (ANB-3066, TP-H1207, TP-H1202, CYH, VRP and TP-N1035)
were completed. Small ¼-inch cubes of propellant were placed in each solvent.
The samples were observed over a 24-hour period with sampling occurring at
the ½, 1, 2, 4, 6, 8 and 24 hour interval. The results of this original
screening are given in Table XI.

Following the initial screening, larger propellant samples were subjected
to the solvents. Potassium hydroxide (KOH) was added to some of the solvents
to evaluate synergistic effects of adding the base to the organic solvent.
The results of these preliminary tests are given in Table XII. These results
are summarized below.

1. ANB-3066 Propellant. Five solvents softened the propellant, making
    it easier to cut. Two solvents, toluene and benzene, also swelled the
    propellant. Addition of potassium hydroxide (KOH) to tetrahydrofuran

    (THF) did not appear to enhance the degradation.

2. TP-H1207. Five solvents softened the propellant. Carbon tetrachloride
    also made the residue sticky which would hinder further processing.
    Addition of KOH to ether (ethyl) did not enhance degradation.

3. TP-H1202. Five solvents softened and swelled the propellant. Addition
    of KOH to THF did not enhance the degradation.

4. CYH. Five solvents degraded the propellant by completely dissolving
    the binder. Addition of KOH to THF produced an exothermic reaction,
    probably decomposition and hydrolysis of the nitroglycerin (NG).

5. VRP. Four solvents indicated degradation by softening and swelling
    the propellant. Addition of KOH and dimethylsulfoxide (DMSO) dissolved
    the binder and produced an exothermic reaction. In addition to the
    probable decomposition of the NG, a potential decompatability between
    sulfur and ammonium perchlorate (AP) may also contribute to the ·
    exothermic reaction.

6. TP-N1035. Four solvents evidenced degradation by softening the pro-
    pellant. Addition of DMSO and KOH dissolved the binder and produced
    an exothermic reaction. The same reactions suggested for VRP propellant
    probably apply for TP-N1035 also.

TABLE XI.   Results of Solvent Screening Tests on Propellants

Solvent	VRP	TP-N 1035	CYH	ANB 3066	TP-H 1207	TP-H 1202	Number of Propellants Affected*
Acetone	-	-	X	-	X	X	3
Acetonitrile	-	-	X	-	X	X	3
Carbontetrachloride	-	-	-	X	X	X	4
Chloroform	-	X	-	X	X	X	4
Cyclohexane	-	-	-	X	X	X	3
DMF	X	X	X	-	X	-	4
DMSO	X	X	X	-	X	-	4
pDioxane	X	X	X	-	X	X	5
Ether	-	-	-	X	X	X	3
Ethyl Acetate	X	X	X	X	-	X	5
Hexane	-	-	-	-	-	X	1
Methanol	-	-	X	-	-	-	1
Methylene Chloride	-	-	-	X	X	X	3
THF	X	X	X	X	-	X	5
Toluene	-	-	-	X	X	X	3
Benzene	-	-	-	X	X	X	3
CS_2	X	-	-	X	-	-	2
Ethylene Glycol	X	X	-	-	-	-	2
Methyl Cyclohexane	X	X	-	-	-	-	2
MEK	-	-	X	X	X	X	4
1 methyl-2 pyridine	-	-	-	-	-	-	0
1-propanol	-	-	-	-	-	-	0
2-propanol	-	-	-	-	-	-	0
Tetrachloroethylene	X	X	-	-	X	-	3
Tetramethylenesulfane	-	-	-	-	-	-	0
Trichloroethylene	-	-	-	-	-	-	0
2,2,4 trimethylpentane	-	X	-	-	X	-	2
M-xylene	-	-	-	X	-	-	1

* This number indicates the number of propellants, of the six tested, which were positively affected by the solvent.

X - indicates a positive affect, softening and/or swelling of the propellant by the solvent.

TABLE XII.   Preliminary Results of Screening Test of
Solvent Degradation of Propellants

Propellant	Class	Principal Ingredients	Potentially Useful Solvents	Results
ANB 3066	1.3	CTPB Polymer, HK-868, NH$_4$ClO$_4$, Aluminum, Polybutene.	Chloroform Cyclohexane Tetrahydrofuran Tetrahydrofuran w/50% KOH Toluene Benzene	Softens propellant. Softens propellant. Softens propellant. Softens propellant, no improvement due to KOH. Softens propellant, propellant swells. Softens propellant, propellant swells.
TP-H1207	1.3	HTPB Polymer, IPDI, NH$_4$ClO$_4$, Aluminum, Tepanol.	Carbon Tetrachloride Chloroform Cyclohexane Toluene Ether Ether w/50% KOH	Softens propellant, sticky. Softens propellant. Softens propellant. Softens propellant. Softens propellant. Softens propellant, no improvement due to KOH.
TP-H1202	1.3	HTPB Polymer, IPDI & ODI, NH$_4$ClO$_4$, Aluminum, HMX, Tepanol.	Carbon Tetrachloride Chloroform Methylene Chloride Toluene Tetrahydrofuran Tetrahydrofuran w/50% KOH	Softens and swells propellant. Softens and swells propellant. Softens and swells propellant. Softens and swells propellant. Softens and swells porpellant. Softens and swells propellant, no change due to KOH.
CYH	1.1	Nitroglycerine, Nitrocellulose, NH$_4$ClO$_4$, Aluminum, Triacetin, Stabilizers.	Acetone Acetonitrile Ethyl Acetate Methanol Tetrahydrofuran	Binder dissolved in 2 hrs., immediate leaching of yellow color. Binder dissolved in 2 hrs., immediate leaching of yellow color. Binder dissolved in 2 hrs., leaching causes yellow coloration of solvent. Binder dissolved in 2 hrs., leaching causes yellow coloration of solvent. Binder dissolved in 2 hrs., leaching causes yellow coloration of solvent.

TABLE XII.
(Continued)

Preliminary Results of Screening Test of
Solvent Degradation of Propellants

Propellant Class		Principal Ingredients	Potentially Useful Solvents	Results
VRP	1.1	Nitroglycerine, Nitrocellulose, H-T Polyester, Aluminum, HMX, Stabilizers.	Tetrahydrofuran w/50% KOH	Binder dissolved in 2 hrs., leaching causes yellow coloration of solvent. Exothermic reaction evident.
			Ethyl Acetate	Swells and softens propellant, leaching causes yellow coloration of solvent.
			Tetrahydrofuran	Swells and softens propellant, leaching causes yellow coloration of solvent.
			Carbon Disulfide	Swells and softens propellant, leaching causes yellow coloration of solvent.
			Dimethylsulfoxide	Swells and softens propellant, leaching causes yellow coloration of solvent.
			Dimethylsulfoxide w/KOH	Binder dissolved in 2 hrs., exothermic reaction evident.
TP-Ni035	1.1	Nitroglycerine, TMETN, Desmodur N-100, NH$_4$ClO$_4$, Aluminum, HMX, PCP, CAB, TPB.	Ethyl Acetate	Softens propellant, leaching causes yellow coloration in solvent.
			P-Dioxane	Softens propellant, leaching causes yellow coloration in solvent.
			Dimethylformamide	Softens propellant, leaching causes yellow coloration in solvent.
			Dimethylsulfoxide	Softens propellant, leaching causes yellow coloration in solvent.
			Dimethylsulfoxide w/KOH	Softens propellant, leaching causes yellow coloration in solvent.
				Binder dissolved in 3 hrs., exothermic reaction.

The samples of the contaminated solvents and propellant residues were tested to determine hazard potential (via safety tests) and composition.

Penetrometer readings were taken after soaking one side of one-inch square cubes of propellant in varying solvents for 24 hours. The cubes were soaked to a depth of 5 mm on one side in solvent and readings were taken at 2.5, 11.0 and 18.0 mm from the base of the side soaked. The results were tabulated in Table XIII. All solvents had a softening effect on all the propellants. Ethyl acetate, a relatively polar solvent, apparently had less softening effect on the TP-H1202, TP-N1035, VRP and ANB-3066 propellants than did THF. THF, in general, affected the propellants the most when in contact and was also absorbed slightly better than ethyl acetate, methylene chloride and cyclohexane. CYH propellant was tested with each solvent and, in all cases, the propellant softened to such a considerable degree that it was impractical to handle and analyze properly.

Penetrometer readings indicate that THF is the solvent that is best absorbed by a majority of the propellants. Ethyl acetate is also a solvent that is absorbed and softens the propellant.

After the propellant was removed from the solvent for the penetrometer tests, the solvent was tested by Fourier Transform Infrared Spectrometry (FTIR) to determine the component(s) of the propellant removed by the solvent. Table XIV lists the ingredients extracted by the various solvents.

Shore A Hardness Test

Procedure. 1 x ¼ x ¼ samples of propellant were placed in 25 mlr of solvent and allowed to soak for 24 hours. The solvent was then decanted from the solid cube of propellant. Shore A testing was done immediately after the solvent was removed and then again 18 hours after air drying at ambient temperature.

TABLE XIII. Penetrometer Readings on Solvent Affected Propellant

Propellant	Before Solvent Reading (mm)	Ethyl Acetate			Methylene Chloride			THF			Cyclohexane		
		Distance From Solvent Soaked Edge of Cube, d**											
		2.5	11	18	2.5	11	18	2.5	11	18	2.5	11	18
TP-H1207	25				67	54	47				107	55	48
TP-H1202	16	100	42	42	103	45	41	260	75	45	58	34	32
ANB 3066	17	75	52	41	50	39	32	351	80	52			
TP-N1035	23	43	37	38				52	46	46			
VRP	27	45	38	40				70	48	49			
CYH	6		*			*			*			*	

* Softened and even though, kept a shape, was too messy to handle.

** Location

Depths of Solvent, 5 mm

2.5 mm
11 mm
18 mm
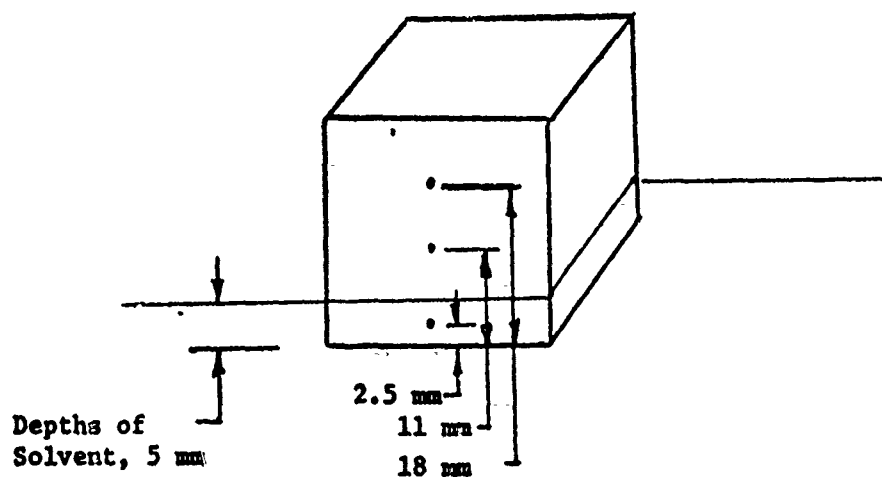
TABLE XIV.   INGREDIENTS EXTRACTED FROM PROPELLANTS BY SOLVENTS

SOLVENT			PROPELLANTS			
	CYH	VRP	TP-H1207	TP-H1202	TP-N1035	ANB-3066
THF	NG, NC PCP	NG 2NDPA PGA	HC Polymer AP	HC Polymer AP HMX	NG	CTPB
DMSO	PCP, NG AP	NG PGA	HC Polymer	HC Polymer	NG PCP	--
Methanol	NG PCP	NG	AP	AP	NC	AP
Benzene	NG	NG	HC Polymer	HC Polymer	NG	CTPB
Acetone	NG, HMX PCP, AP Triacetin	NG PGA HMX	AP	AP HC Polymer	NG HMX	AP CTPB
Ether	NG PCP	NG PGA	HC Polymer	HC Polymer	NG	AP
DMF	NG	--	--	--	NG HMX	--
Ethyl Acetate	NG PCP	NG 2NDPA	HC Polymer	HMX HC Polymer	NG TMETN PCP HMX	CTPB
Methylene Chloride	NG PCP HMX	NG	HC Polymer	HC Polymer	NG HMX	CTPB

Shore A hardness tests (using nine different solvents) on the six different propellants indicated that THF, methylene chloride and ethyl acetate were solvents which softened the propellants best (See Table XV). Ether appeared to harden the CYH, VRP and TP-N1305 propellants.

Results of safety tests on the solvents, containing the propellant extractables and on the propellant residues, are summarized in Table XVI. Safety tests on the propellant residues are given in Table XVII.
It has been reported that aqueous ammonium hydroxide is very effective in degrading the polyurethane binder system in propellants. TP-H1202, TP-H1207 and ANB-3066 propellants were subjected to 1 N. aqueous ammonia for two weeks. The effect on the propellant was minimal and comparable to the effect of soaking in water. The propellant was slightly swollen and the AP dissolved as occurs in water. No further use of this reagent is recommended.

Specific tests were conducted on propellants containing nitroglycerin (NG), nitro-cellulose (NC) and HMX to identify practical means for deactivating these constituents or, in the case of HMX, for dissolution from the propellant and for recovery. The results were as follows:

Thirty grams of VRP and TP-N1035 were placed in 100 mls of five different solvents. Samples were taken at various times and analyzed by HPLC for HMX content. Samples were also analyzed for nitroglycerin leaching by IR analysis.

Table XVIII lists the results of HMX leaching from CYH, VRP and TP-N1035 propellant. Acetone appeared to dissolve HMX the best with MEK being the next best solvent. Cyclohexane in all three cases was a very poor solvating agent. These results match closely with the polarity of the solvents as would be expected. Acetone being the more polar solvent, should solvate polar compounds better than nonpolar solvents like cyclohexane. See Table XIX for polarity data on the solvents.

The degree of leaching of nitroglycerin from VRP, CYH and TP-N1035 using acetone, MEK, ethyl acetate, THF and cyclohexane can also be correlated with the polarity index of these solvents. Acetone, MEK, ethyl acetate and THF all have large

TABLE XV. COMPARISON OF SOLVENT EFFECTS ON SHORE A HARDNESS ON PROPELLANTS

TWR-30684

Solvent	CYH		VRP		Propellant TP-H1207		TP-H1202		TP-N1035		ANB-3066	
	0 Hours	18 Hours	0 Hours	18 Hours	0 Hours	18 Hours	0 Hours	18 Hours	0 Hours	18 Hours	0 Hours	18 Hours
Control (No Solvent)	79	—	47	—	55	—	68	—	49	—	73	—
THF	*	*	2.5	22	0	0	0	0	2.5	31	0	0
DMSO	*	*	4	6	44	39	54	60	24	35	0	0
Methanol	*	*	33	49	38	40	47	52	51	76	41	41
Acetone	*	*	9	23	17	18	20	20	11	53	18	23
Ether	96	98	61	77	0	14	5	24	64	85	4.5	29
DMF	*	*	0	7	37	35	41	42	27	64	0	0
Ethyl Acetate	*	*	15	35	9	13	13	23	21	47	15	32
Methylene Chloride	91	93	6	37	0	0	0	18	2	34	0	31
Cyclohexane	83	83	46	45	4	14	15	27	49	~50	13	32

* = Liquid - too soft to test.

TABLE XVI. HAZARD INFORMATION ON SOLVENT EXTRACT FROM PROPELLANT SOAK

TWR-30684

Sample	TC Indirect Impact (Inches)	ABL Impact (cm)	TC Strip Friction (Pounds)	ABL Sliding Friction (8 ft/sec)	TCESD Joules	ABL ESD (Joules)	TC Autoignition 300°F (Hours)	TC Autoignition 225°F (Hours)	Explosive Class
VRP									
THF	>46	80	*	*	*	*	*	*	GL
Ethyl Acetate	>46	80	>64	800	<.500	<.075	*	*	GL
CYH									
Acetonitrile	>46	64	*	50	*	*	*	*	YL
Acetone	>46	80	*	100	*	*	*	*	GL
Methanol	>46	64	>64	180	6.83	.075	24	*	YL
TP-N1035									
p-Dioxane	>46	80	>64	800	<.05	.06	*	*	GL
DMF	>46	80	>64	80	>8	1.25	.79	*	YL
Ethyl Acetate	>46	80	*	*	*	*	*	*	GL
TP-H1202									
Carbon Tetrachloride	>46	80	>64	800	>8	1.25	*	*	GL
TP-H1207									
Cyclohexane	>46	80	>64	420	>8	<.06	*	*	GL
Carbon Tetrachloride	>46	80	>64	800	>8	1.25	*	*	GL
ANB-3066									
Cyclohexane	>46	80	>64	420	>8	.06	*	*	GL
Chloroform	>46	80	>64	420	>8	.625	*	*	GL

* Did not complete the test.

TABLE XVII. HAZARD INFORMATION ON PROPELLANT RESIDUES AFTER SOLVENT SOAK

TWR-30684

Sample	TC Indirect Impact (Inches)	ABL Impact (cm)	TC Strip Friction (Pounds)	ABL Sliding Friction (8 ft/sec)	TCESD Joules	ABL ESD (Joules)	TC Autoignition 300°F (Hours)	TC Autoignition 225°F (Hours)	Explosive Class
VRP									
After THF Soak	9.91	41	> 64	180	> 8	6.25	.72 (Burn)	24 No ignition	YL
After Ethyl Acetate Soak	11.80	41	> 64	180	> 8	6.25	.48 (Burn)	24 No ignition	YL
CYH									
After Methanol Soak	10.60	17	39.20	100	> 8	6.25	.63 (Burn)	24 No ignition	YL
After Ethyl Acetate Soak	43.80	11	> 64	50	> 8	6.25	.32 (Burn)	24 No ignition	YL
TP-N1035									
After DMSO Soak	42.56	17	63.67	800	> 8	1.25	.38 (Burn)	24 No ignition	YL
After DMF Soak	46	33	> 64	800	> 8	1.25	--	24 No ignition	GL
After Ethyl Acetate Soak	16.20	17	> 64	660	8	6.25	1.1 (Burn)	24 No ignition	YL
TP-H1202									
After THF Soak	13.80	17	> 64	50	> 8	1.25	--	24 No ignition	YL
After Methylene Chloride Soak	15.18	17	60.40	50	> 8	6.25	--	24 No ignition	YL
TP-H1207									
After Cyclohexane Saok	17.18	21	63.00	100	8	6.25	--	24 No ignition	GL
After Ether Soak	17.40	21	62.20	50	8	1.25	--	24 No ignition	YL
ANB-3066									
After THF Soak	13.00	21	> 64	180	> 8	6.25	--	24 No ignition	GL
After Cyclohexane Soak	16.40	21	63.60	100	> 8	6.25	--	24 No ignition	GL

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TABLE XVIII. HMX LEACHING FROM PROPELLANT

A. VRP

Solvent	1 Hour	2 Hours	4 Hours	8 Hours	24 Hours	48 Hours
Ethyl Acetate						
mg/ml	0.51	0.85	1.04	1.03	1.01	--
%	9	8	7	6	4	--
Acetone						
mg/ml	2.4	3.6	5.1	7.4	10.4	--
%	23	24	22	18	18	--
Methyl Ethyl Ketone						
mg/ml	1.7	2.1	3.2	3.6	4.0	--
%	21	20	20	16	12	--
THF						
mg/ml	0.9	1.1	1.5	1.5	1.7	1.7
%	4	5	6	5	4	3
Cyclohexane						
mg/ml	0	0	0	0	0	0
%	0	0	0	0	0	0

B. TP-N1035

	2 Hours	4 Hours	24 Hours
Ethyl Acetate			
mg/ml	0.9	1.5	1.9
%	6	7	5
Acetone			
mg/ml	7.7	12.0	17.3
%	26	28	25
Methyl Ethyl Ketone			
mg/ml	3.6	5.9	8.2
%	17	17	15
THF			
mg/ml	1.2	1.6	2.7
%	3	3	3
Cyclohexane			
mg/ml	0.02	0.001	0.02
%	0.6	0.04	0.3

C. CYH

	1 Hour	2 Hours	4 Hours	8 Hours	24 Hours
Ethyl Acetate					
mg/ml	1.2	1.7	3.1	2.5	2.0
%	5	5	6	4	3
THF					
mg/ml	0.86	1.7	2.8	2.6	2.9
%	4	4	5	4	4
Cyclohexane					
mg/ml	0	0	0	0	0.004
%	0	0	0	0	0.13

TABLE XIX. POLARITY INDEX OF SELECTED SOLVENTS

Polarity Index	Solvent
6.6	Methanol
6.5	DMSO
5.4	Acetane
5.4	Ethylene glycol
4.5	MEK
4.3	Chloroform
4.3	Ethyl Acetate
4.2	THF
3.4	Methylene chloride
3.0	Benzene
2.9	Ethyl Ether
2.3	Toluene
1.0	Carbon Disulfide
0.0	Cyclohexane

polarity index numbers and by inspection of the percent nitroglycerin extracted appear to extract nitroglycerin quite readily. Cyclohexane with a low polarity index number does not extract nitroglycerin very readily.

The approximate rates of leaching of nitroglycerin from VRP, TP-N1035 and CYH propellants are given in Table XX.

Solvent extracts of CYH, VRP and TP-N1035 propellant were subjected to various hydrolysis and reducing conditions. The degradation of not only nitroglycerin but also that of other nitro containing compounds (nitrocellulose in CYH) was followed by IR analysis.

Preliminary results indicate that the nitro containing compounds can be degraded using various concentrations of ethanol amine and sodium hydroxide solutions. TP-N1035 ethyl acetate extract appears to be degraded quicker with ethanol amine as catalyst than with 1.0 N sodium hydroxide solution. This is probably due to the different solubilities of the basic solutions in ethyl acetate.

Based on the results above, it is concluded that solvents can be desensitized or degraded to assist in removing the propellant from the case. Whether these solvents can be incorporated into an ingredient recovery scheme is beyond the scope of this program. The initial indication is that this could be a useful purpose for using solvents; however, due to the high risk of damage to the case, it is doubtful that this method should be recommended for case salvage operations.

TABLE XX.   NITROGLYCERIN LEACHING FROM PROPELLANT

A. VRP

Solvent	Time				
	1 Hour	2 Hours	4 Hours	8 Hours	24 Hours
Ethyl Acetate					
mg/ml NG	10.6	15.8	24.1	29.0	46.1
% NG	85	78	88	85	88
Acetone					
mg/ml NG	13.5	18.2	27.3	51.6	69.2
% NG	60	62	59	61	58
Methyl Ethyl Ketone					
mg/ml NG	12.5	18.8	24.4	35.7	54.6
% NG	82	81	76	72	78
THF					
mg/ml NG	12.7	16.9	22.6	24.7	49.0*
% NG	30	40	49	36	56
Cyclohexane					
mg/ml NG	0.6	0.8	1.2	1.5	1.9
% NG	--	--	--	--	--

* THF samples: 48 hrs., 53.8 mg/ml; 72 hours, 52.8 mg/ml.

B. TP-N1035

	2 Hours	4 Hours	24 Hours
Ethyl Acetate			
mg/ml	10.8	16.8	29.0
%	64	68	71
Acetone			
mg/ml	11.4	17.0	28.9
%	39	43	42
Methyl Ethyl Ketone			
mg/ml	11.9	17.5	31.5
%	57	49	59
THF			
mg/ml	9.6	13.2	28.9
%	28	26	37
Cyclohexane			
mg/ml	0.74	0.98	2.1
%	7	16	9

C. CYH

	1 Hour	2 Hours	4 Hours	8 Hours	24 Hours
Ethyl Acetate					
mg/ml	15.3	20.7	29.0	33.7	35.5
%	60	58	56	58	57
THF					
mg/ml	9.1	16.1	20.8	22.7	27.4
%	37	27	28	32	34
Cyclohexane					
mg/ml	0.34	0.50	0.71	1.11	1.67
%	8	16	29	44	58

6.2 Effect of Solvent on the Propellant/Liner Bond Strength

Three propellant/liner/insulation systems were evaluated to assess the effects
of the selected solvent(s) upon the rebonding of each system. Each system
was evaluated with bond specimens that are regularly used for the respective
bond systems. These systems, and the selected solvents are as follows.

Program	Bond System	Selected Solvent
MX	TP-H1207/UF-2186/EPDM	Cyclohexane
Stage III Minuteman	ANB-3066/SD-851-2/V45	Cyclohexane
C4	VRP/Powder Embedment/EPDM	Ethyl Acetate

Bond specimens (90° peel and bond-in-tension) were fabricated for each of
the three systems. For direct comparison purposes, specimens were made
with insulation that had not been exposed to solvent as well as insulation
that had been exposed to solvent for 24 hours. After solvent exposure, the
insulation was prepared for lining as dictated by the production process
for each system.

The test results for all the bond systems are complete and indicate
no detrimental effects of a solvent soak upon the bond. These data are
shown below.

Program System	Bond Strength Test Results			
	90° Peel (pli)		Bond-in-Tension (psi)	
	Control	Solvent Soaked	Control	Solvent Soaked
MX	13.0	13.5	117	110
	12.0	11.5	124	117
	8.0	7.5	-	-
Average	11.0	10.8	120	114
C4	7.0	7.2	107	113
	7.4	6.8	109	116
	-	-	110	117
	-	-	108	114
	-	-	105	123
Average	7.2	7.0	108	117
Stage III	8.8	11.7	68.4	81.5
	17.6	10.0	76.9	62.7
	12.0	18.3	66.0	74.4
Average	12.8	13.3	70.4	72.9

It is concluded based upon these results that, in general, no detrimental effects on the propellant/liner/insulation system would be expected. Testing of the specific system and the solvent selected for propellant removal would be necessary prior to starting a large case salvage program.

These results and conclusions appear to be academic. While the use of solvents does not appear to affect the relining and reloading of the motor, the use of solvents has already been ruled out due to the high risk of damage to the case by the solvent.

6.3 Effect of Solvents on the Insulation

If solvents are used during the removal of the propellant, the insulation will be exposed to the solvent for indeterminate periods of time. The following tests were performed to determine whether permanent, detrimental changes would occur to inhibit the reuse of the insulation.

The two insulations selected for testing were EPDM-053A and V-45. EPDM is currently used in the MX first stage motor and V-45 was used in the Minuteman III third stage motor.

6.3.1 Screening Tests

One-inch squares, cut from an MX Kevlar case with EPDM insulation in tact, were subjected to each solvents for a 16-hour period. After air drying for 16 hours at ambient temperature, the samples were visually investigated and the results indicated in Table XXI. The Kevlar case material was affected greatly by most of the solvents examined. This may be due to the fact that the case was cut, allowing a greater surface area than normal to contact the solvent. Further testing was considered which would circumvent this problem of surface area soaking.

It is apparent from these results that for each case salvage operation, the specific insulation in the case must be tested with the specific solvent to be used. With EPDM, of the 16 solvents tested, only cyclohexane, toluene, benzene, ethyl ether and carbon disulfide observably produced immediate changes in the rubber. Whether the swelling produced permanent effects was not determined in these tests but results of swell tests reported later do indicate permanent effects. Perhaps the most important result was that in seven examples, the rubber to case bond was weakened or destroyed causing separation of the insulation from the case resin.

6.3.2 Case (Glass) and V45 Insulation Solvent Soak

One-inch squares of glass case and V45 insulation were taken directly from a fired Minuteman case. These samples were soaked in fourteen different solvents for a twenty-four hour period. After the soaking, the samples were dried at ambient temperature under vacuum for eighteen hours. This

TABLE XXI.   Kevlar Case/EPDM Solvent Affect

Solvent	Kevlar Case	EPDM
Chloroform	Deteriorates Epoxy Resin (DER)	Separates from Kevlar (SK)
DMF	DER	SK
Carbon Tetrachloride	DER	SK
Methanol	No apparent resin deterioration.	No SK
Acetone	No apparent resin deterioration.	No SK
Cyclohexane	No apparent resin deterioration.	Swollen/No SK
Ethyl Acetate	DER	No SK
THF	DER	SK
Toluene	Some resin deterioration.	Swollen/SK
Benzene	Some resin deterioration.	Swollen/SK
Ethyl Ether	Some resin deterioration.	Slight swelling/slight separation.
Methylene Chloride	DER	No SK
Acetonitrile	No apparent resin deterioration.	No SK
Carbon Disulfide	Some resin deterioration.	Swollen/SK
DMSO	Some resin deterioration.	No SK
p-Dioxane	DER	No SK

drying period was not sufficient and an additional drying period was conducted at 53°C (under vacuum) for twenty-four hours. Weights taken at this time indicate that, with some solvents, the samples were still not dry.

Table XXII gives the qualitative results of the effects the solvents had on the case and V45 insulation materials after twenty-four hours of soaking. In all cases (except that of methylene chloride) the glass case material was, by visual observations, not adversly affected. With methylene chloride, however, the case material separated between the two differently wound sections. The outer epoxy layer of the case flaked off from the glass in the cases of THF, methanol, chloroform, methylene chloride, DMF and benzene soaking. Only the cyclohexane, methanol, DMSO and carbon disulfide had little effect on the swelling of the V45 insulation. This information matches well with the swell index data obtained on V45 insulator which follows.

6.3.3   Swell Tests on EPDM and V45 Rubber

The effect of different solvents on EPDM and V45 rubbers was determined using the procedure outlined in DAP-0237, Revision A. Basically, the steps are the following.

1.   A specimen about 3/8" in diameter was weighed and soaked in the solvent for about 24 hours.

2.   The samples were removed from the solvent, immediately put in a tared weighing bottle and reweighed.

3.   The samples were dried in a vacuum oven at about 65°C and then reweighed.

The swelling index is calculated by:

$$\text{Swelling Index} = \frac{\text{Swollen Weight}\quad\text{(Before drying step)}}{\text{Final Dried Weight}\quad\text{(after drying)}}$$

The percent extract is calculated by:

$$\% \text{ Extract} = \frac{\text{Weight of original sample -- final dried weight}}{\text{Weight of original sample}} \times 100$$

TABLE XXII. STAGE III MINUTEMAN CASE MATERIAL/SOLVENT SOAK RESULTS   TWR-30684

Solvent	Case Material			V45 Insulation	
	No Apparent Effect	Outer Epoxy Layer Flake	Swollen	Separation from Case	Color Leach Into Solvent
Cyclohexane	x	-	-	-	-
Ethyl Acetate	x	-	x	x	Red/Amber
THF	x	x	x	x	Light Amber
Acetone	x	-	x	x	Light Amber
Methanol	x	x	-	-	-
Chloroform	x	x	x	x	Light Amber
Acetonitrile	x	-	x	x	Light Amber
Methylene Chloride	Case Material Separation	x	x	x	Light Amber
Ether	x	-	Slight	x	Light Amber
DMSO	x	-	x	-	Light Amber
DMF	x	x	x	x	Light Amber
Toluene	x	-	x	x	Light Amber
Benzene	x	x	x	x	Light Amber
Carbon Disulfide	x	-	-	-	Light Amber

x Indicates Yes
- Indicates No

From the data summarized in Table XXIII obtained on the V-45 rubber, cyclohexane, methanol and DMSO were the solvents which gave the least amount of swelling with percent extracted number of 2.13, 4.22 and 5.14, respectively. The six other solvents had percent extracted numbers greater than 9%. Percent extracted data on the EPDM 053A insulation indicates that DMSO, methanol, DMF and acetone gave the least amount of swelling with extracted numbers less than 5%. The other five solvents had percent extracted numbers ranging from 6% to 11%. In general, the EPDM 053A insulation was effected by the solvents less than the V-45 insulation.

The important information to access in Table XXIII is that of the percent of extracted material from the insulation and the volume of solvent absorbed by the insulation. The V-45 (control) insulation absorbed a large amount of methylene chloride, THF and cyclohexane and a small amount of acetone, methanol and ethyl acetate. The V-45 (case) insulation absorbed a large amount of methylene chloride, TFH and DMF and very little ether, methanol and DMSO. EPDM-053A also absorbed a large amount of cyclohexane and THF like the V-45 control and a small amount of acetone and methanol. In all three cases of insulation, THF was absorbed to a great extent which in turn will swell the insulation which may cause separation of the insulation from the case material. THF and ethyl acetate both appeared to extract a large amount of materials from the V45 case and control samples. Analysis of the extract indicates that dioctyl phthalate and NBR copolymer (butadiene/acrylonitrile) were some of the materials removed from the insulation. For the EPDM 053A, THF and cyclohexane were solvents which extracted the most materials from the insulation.

Swell and percent extracted indicate acetone, methanol and ethyl acetate affect V45 (control) the least; ether, methanol and DMSO affect V45 (case) the least; acetone and methanol affect EPDM 053A the least.

TABLE XXIII.   Solvent Swell Data on V45 (Case), V45 (Control),
                 EPDM 053A Insulation

	Methanol	Cyclo-hexane	DMSO	Methylene Chloride	Acetone	DMF	Ether	THF	Ethyl Acetate
V45 (Control)									
Swell Index	1.2	1.3	1.8	6.0	2.5	3.6	1.3	4.0	2.5
% Extracted	5.5	5.8	6.7	12.0	14.0	14.0	14.0	15.0	15.0
Volume of Solvent Absorbed, ml	.04	.55	.16	.77	.34	.52	.42	.64	.29
V45 (Case)									
Swell Index	1.2	1.2	1.7	6.5	2.6	3.5	1.4	4.0	2.6
% Extracted	4.2	2.1	5.1	13.5	12.2	9.6	11.6	11.5	12.4
Volume of Solvent Absorbed, ml	.05	.10	.17	1.04	.53	.80	.08	1.00	.46
EPDM 053A									
Swell Index	1.1	3.6	1.3	2.3	1.3	1.3	1.9	3.2	1.3
% Extracted	2.2	9.5	1.8	8.9	4.6	3.4	9.9	11.0	6.6
Volume of Solvent Absorbed, ml	.04	.58	.06	.16	.06	.04	.16	.41	.23

Definitions

Swell Index = $\dfrac{\text{swollen weight}}{\text{fixed dried weight}}$

% Extracted = 100X $\dfrac{\text{wt of original sample} - \text{final dried weight}}{\text{wt of original sample}}$

Volume of Solvent, ml = (wt of wet sample − wt of dry sample) x density

Samples of two insulation materials, EPDM-053A and V45, were subjected to
solvents for a period of 24 hours and then, at ambient temperature, dried
in a vacuum for 18 hours. The samples of V45 were obtained from two sources;
one was fresh stock (control) and the other (case) was V45 rubber stripped
from a third stage Minuteman case by the heat and peel method. The mechanical
properties of the rubber was measured to determine whether the exposure to
the various solvents had affected the mechanical properties of the insulation.
The results are tabulated in Table XXIV.

Tensile, elongation, modulus and Shore A information indicate that for EPDM
053A, ether, methylene chloride and THF had the least affect. THF and
cyclohexane were solvents with the least offset on V45 (case) insulation and
THF, ethyl acetate and acetone had the least affect on V45 (control) insulation.

For the EPDM 053A insulation, ether, methylene chloride and THF had the
least affect while cyclohexane had the most affect. The percent elongation
of the insulation increased with over half of the solvents investigated
indicating the possibility that solvent was still present in the sample and/or
a loss of materials. Percent extracted data, Table XXIII indicates that the
high strain of the DMSO sample may be due to solvent still present since
the percent extracted was only 1.8%. The change in elongation caused by
THF is possibly explained by a loss of materials (11% extracted).

Only ether and ethyl acetate appeared to harden the insulation. The other
solvent systems did not appreciably effect the Shore A hardness of the
insulation. The increased hardness may be due to the removal of the
plasticizer.

The V45 (case) material was obtained directly from a Minuteman III case.
The solvents which effected the insulation the most were DMF and DMSO while
THF and cyclohexane appeared to have the least effect. Once again, the
elongation of the insulation was affected as with the EPDM 053A material;
however, in this case, the percent elongation, strain was decreased. Percent

TABLE XXIV. EFFECTS OF SOLVENTS ON THE MECHANICAL PROPERTIES OF INSULATION

A.   EPDM 053A

Stock: EPDM/CR-FB/HiSil 233
Cure: 300°F x 150' in press

Solvent	Tensile (psi)	Elongation (%)	Modulus (psi)	Shore A (zero time/15 sec)
Control	2017	675	299	67/57
Ether	2066	663	312	70/62
Cyclohexane	1783	646	248	.67/55
DMF	919	688	271	62/54
Ethyl Acetate	1914	642	298	70/58
Acetone	1860	701	266	67/56
Methanol	1873	746	251	66/52
Methylene Chloride	2021	726	266	68/60
DMSO	1988	726	274	66/55
THF	2136	752	284	284 64/52

TABLE XXIV (CONTINUED)   TWR-30684

V45 From MM III Case

B.

Stock: NBR/HiSil 233
Cure: Production Cycle

Solvent	Tensile (psi)	Elongation (%)	Modulus (psi)	Shore A (zero time/15 sec) smooth/rough
Control	2325	600	398	67/56/66/54
Ether	2193	560	397	73/52/71/55
Cyclohexane	2003	590	330	57/48/56/44
DMF	1485	510	305	43/37/42/36
Ethyl Acetate	2105	540	400	65/50/64/48
Acetone	2155	510	410	69/56/64/52
Methanol	2070	550	378	66/50/61/50
Methylene Chloride	2090	510	418	66/46/63/50
DMSO	1585	551	299	49/40/49/39
THF	2238	540	907	59/50/60/51

TABLE XXIV (CONTINUED)   TWR-30684

V45 (Control)

Stock: NBR/HiSil 233
Cure: 300°F x 150' in press

Solvent	Tensile (psi)	Elongation (%)	Modulus (psi)	Shore A (zero time/15 sec)
Control	2733	591	452	69/50
Ether	2785	576	473	74/59
Cyclohexane	2308	584	395	59/45
DMF	2578	595	427	63/50
Ethyl Acetate	2776	596	459	75/58
Acetone	2734	603	455	74/58
Methanol	2684	623	431	72/55
Methylene Chloride	2956	595	498	76/58
DMSO	2476	637	362	56/41
THF	2780	600	454	72/58

extracted information indicated more materials were extracted from the V45
(case) than the EPDM. Thus, a greater effect on the physical properties of
the insulation.

Shore A information indicates that in the case of DMF and DMSO, a large
negative change in hardness has occurred. This change may be due to the
incomplete stripping of the solvents and the residue then acting as
plasticizer or since a material was extracted from the sample, a change
in rheology. There were apparently two different cures or batches of
materials used in the insulation and both surfaces were tested. There
was no major difference in Shore A measurements. There was an increase
in hardness after exposure to ether. This increase may be due to the
removal of DOP plasticizer.

The solvents which affected the V45 (control) the most were DMF, DMSO and
cyclohexane while THF, ethyl acetate and acetone had the least affect. The
difference between the control and case V45 is probably due to the aging of
the insulation.

It is interesting to note that the percent extracted material from the V45
(control) was greater than that from the V45 (case) samples which might
explain the increase hardness of the V45 (control) and not that of the V45
(case).

Tensile bars (dog bones) made of EPDM 053A insulation were soaked for a twenty-four hour period in samples of methylene chloride and THF with propellant extract. The samples were then removed from the solvent and dried under a vacuum at ambient temperature for twenty-four hours. The mechanical properties of the insulation are listed in Table XXV.

The extract of propellant in either methylene chloride or THF, containing extracted materials from the propellants, did not appear to appreciably affect the tensile strength or elongation percent of the insulation. This indicates that the materials being leached from the propellant do not adversely affect the insulation any more than the plain solvent affect and the insulation.

We conclude from the above results that the extended use of solvents for propellant removal generally would have deleterious effects on the insulation and would not be recommended.

TABLE XXV. MECHANICAL PROPERTIES OF INSULATION SOAKED WITH
SOLVENTS AND EXTRACTABLES FROM THE PROPELLANT

A. EPDM 053A Methylene Chloride – Propellant Soak

Propellant	Tensile (psi)	Elongation (%)	Modulus (psi)	Shore A (0 sec/15 sec)
Control (No Propellant)	2027	.644	315	64/56
TP-H1202	1992	655	304	67/57
TP-H1207	1758	655	268	68/56
ANB-3066	1904	697	273	66/56
TP-N1035	1905	701	272	67/55
VRP	2077	697	298	65/55
CYH	1960	670	293	66/57

B. EPDM 053A THF – Propellant Soak

Propellant	Tensile (psi)	Elongation (%)	Modulus (psi)	Shore A (0 sec/15 sec)
Control (No Propellant)	1993	682	292	65/55
TP-H1202	1690	633	267	66/55
TP-H1207	1872	658	284	65/55
ANB-3066	1872	697	268	64/55
TP-N1035	1854	668	278	64/54
VRP	1817	677	268	64/54
CYH	1740	690	252	64/52

6.4   Effect of Solvents on the Case Materials

The results of preliminary tests on the effect of solvents upon the Kevlar
cases is given in Table XXVI. These results show that most of the solvents
affect the resin system and many of them produce debonding between the
Kevlar and the EPDM insulation. The ranking of the desirability for the
solvents for propellant removal is included to show the justification for
the solvents tested.

The test matrix to evaluate quantitatively the effect of solvents on the
composite case materials was expanded from that listed in the program plan.
The new matrix is shown in Table XXVII. Additional 5.75" bottles, both
Kevlar and glass, were fabricated to allow for testing more conditions and
additional solvents. Samples were fabricated for testing the glass transition
temperature by rheometric dynamic spectroscopy (RDS). This test measures
the chemical degradation of the resin systems whereas the hydrotest bursting
of the 5.75" bottles measures the attack on the integrity of the composite
case structure. The NOL ring was used instead of individual short shear
beam samples to eliminate the diffusion of the solvent into the cut ends.
The entire ring is subjected to the solvent then short shear beam samples
are cut from the ring following the exposure. The short shear beam tests
measure the effect of the solvent on the mechanical properties of the fiber.
Descriptions of the tests and sketches of the NOL rings and short shear beam
specimen are included in Appendix B.

The results of each of these series of tests follows. The two systems used
in these tests, Kevlar/UF-3283/EPDM and Glass/UF-3205/V45, were felt to be
representative, although not exact of two systems which could be of future
interest, MX and Third Stage Minuteman III cases.

TABLE XXVI. Results of Preliminary Testing of Solvents Effect on Kevlar Cases with EPDM Insulation

Propellant	Safety	Solvent	Rank For Propellant Removal	Solvent Effect on Case Specimen	
				Kevlar and Resin	Insulation (EPDM)
VRP	X	DMSO	-	Fibers separate under pressure	No apparent effect
	OK	Ethyl Acetate	1	Dissolves resin	No apparent effect
	OK	THF	2	Dissolves resin	Rubber debonds from case
	X	CS_2	-	Dissolves resin	Rubber debonds from case, swells
TP-H1035	X	DMSO	3	Fibers separate under pressure	No apparent effect
	OK	Ethyl Acetate	1	Dissolves resin	No apparent effect
	X	DMF	2	Dissolves resin	Rubber debonds from case
	X	pDioxane	-	Dissolves resin	No apparent effect
TP-H1207	X	CCl_4	-	Fibers separate under pressure	No apparent effect
	X	$CHCl_3$	-	Dissolves resin	Rubber debonds from case
	X	Cyclohexane	1	No apparent effect	No debonding, rubber swells
	OK	Ether	2	Fibers separate slightly	Rubber debonds from case, swells
	X	Toluene	-	Fibers separate under pressure	rubber debonds from case, swells
TP-H1202	X	CCl_4	-	Fibers separate under pressure	Rubber debonds when pulled
	X	$CHCl_3$	-	Dissolves resin	Rubber debonds from case
	OK	THF	2	Dissolves resin	Rubber debonds from case
	OK	CH_2CL_2	1	Dissolves resin	No apparent effect
	X	Toluene	-	Dissolves resin	Rubber debonds from case, swells
CYH	OK	Acetone	1	No apparent effect	No apparent effect
	OK	Ethyl Acetate	3	Dissolves resin	No apparent effect
	OK	THF	-	Dissolves resin	Rubber debonds from case
	OK	Methanol	2	No apparent effect	No apparent effect
	X	Acetonitrile	-	No apparent effect	No apparent effect
ANB 3066	X	Benzene	-	Fibers separate under pressure	Rubber debonds, swells
	X	$CHCl_3$	-	Dissolves resin	Rubber debonds from case
	X	Cyclohexane	1	No apparent effect	No debonding, rubber swells
	OK	THF	2	Dissolves resin	Rubber debonds from case
	X	Toluene	3	Dissolves resin	Rubber debonds from case, swells

*: X - Indicates restricted use of solvent for carcino-genic or other toxic effects.

OK - Indicates not on OSHA list, but does not preclude exclusion due to flammability.

TABLE XXVII.   Summary of Samples and Tests for Determination
of the Effect of Solvent on Case Materials

Type of Chamber Fiber/Resin/Insulation	Type of Sample	Number of Samples Tested With Solvent**			Control Samples	Test Performance
		Solvent 1	Solvent 2	Solvent 3		
Kevlar/UF-3283/EPDM	5.75" bottle	6*	3	0	3	Hydroburst (chamber integrity)
Glass/UF-3205/V45	5.75" bottle	3	6*	0	3	Hydroburst (chamber integrity)
Kevlar/UF-3283/EPDM	NOL Rings	1	1	1	1	Ten Short-Shear Beam (Mech. Properties)
Glass/UF-3205/V45	NOL Rings	1	1	1	1	Ten Short-Shear Beam (Mech. Properties)
Kevlar/UF-3283/EPDM	RDS Specimen	2*	2*	2*	1	Glass Transition Temperature (chemical degradation of resin)
Glass/UF-3205/V45	RDS Specimen	2*	2*	2*	1	Glass Transition Temperature (chemical degradation of resin)

*   Duplicate tests with samples wet and dried after solvent contact.

**   Solvent 1 = cyclohexane

   Solvent 2 = ethyl acetate

   Solvent 3 = tetrahydrofuran (THF)

6.4.1   <u>Integrity of the Case - 5.75" Bottle Tests</u>

The objective of the bottle burst test is to determine if the solvent
selected for propellant solution will migrate through the rubber
insulation and degrade the resin matrix - fiber reinforced composite case.

5.75" diameter bottles that represent a subscale motor were wound. The
quantity was   expanded from 12 to 30 to allow for testing more solvent
systems. These bottles have been fabricated.

Quantity: 15 bottles, S-901 glass/wet wound UF-3205 resin systems (3rd
            Stage Minuteman)
            15 bottles, Kevlar 49/UF-3283 prepreg (First Stage MX)

Cure Cycle: Glass bottles/2½ hours at 212°F, 2½ hours at 256°F, 2½ hours
             at 312°F
             Kevlar bottles/8 hours at 210°F

Of these 15 bottles/material group, three bottles are burst to establish
pressure at failure. Then 80% of this burst pressure is calculated and the
remaining 12 bottles are hydroproofed at this pressure. Of the remaining
12, three will be controls leaving nine for solvent exposure. Three bottles
per set, therefore, three solvents. After exposure to the solvents, the
bottles were be dried in a vacuum oven
and subsequently burst along with the three control bottles. By
comparison with the control bottles, the solvent exposed bottles may or may
not demonstrate a decreased burst pressure due to the effects of solvent on
the composite due to migration. Being that the interior of the bottle will
be exposed to the solvent (although migration may be extensive enough for
radial migration into outer hoop layers), the bottle is designed for a polar
burst. (For both glass and Kevlar bottles the first two plies that are wound
over the molds are polars.)

The calculations that shall be used in the calculations are as follows:

(Fiber Stress) $\quad \sigma_{hoop} = \dfrac{PR}{t_{hoops}} (1 + \epsilon_o)(1 - \dfrac{\tan^2\theta}{2})$

(Fiber Stress) $\quad \sigma_{polar} = \dfrac{PR}{2 \, t \, polars \, \cos^2\theta} (1 + \epsilon_o)$

Where: $\quad P$ = case burst pressure

$\qquad R$ = case radius at burst (take I.D./2)

$\qquad \theta$ = polar wind angle

$\qquad \epsilon_o$ = failure strain of fiber (unique to each material)

$\qquad t$ = thickness

$\qquad \sigma$ = fiber stress

Interpretation of Data - For the glass bottles, a lower pressure at burst will mean a lower fiber stress by the equations. However, for the Kevlar bottles, it is known that when transverse bonding (perpendicular to direction of fiber) is removed, the fiber stress can be increased. The solvent may decrease this transverse bonding between fiber - resin interface allowing slippage of fibers and could, theoretically, increase the fiber stress. This would not, however, lead to false data interpretation because the decreased transverse fiber-resin interfacial bonding would be apparent in decreased shear strength and probably a lowering of the glass transition temperature. The above remarks are theoretical and will be demonstrated when the data is returned.

Bottle Information: Diameter - 5.75"

         # of polar plies - 2

         # of hoop plies - 5   S-901 glass/V45 Ins.

         # of polar plies - 2

         # of hoop plies - 4   Kevlar/EPDM Ins.

         Resin System UF-3205 (glass) (wet wound)

         Resin system UF-3283 (Kevlar) (prepreg)

         Winding tension - 5 pounds (glass)

         Winding tension - 10 pounds (Kevlar)

         Stress Ratio $\sigma p / \sigma h$ = 1.135 (both types)

Building: M-9

Winding Machine: Tumble Winder, M-10

Hydroburst Pressure Rate: 5000 psi/minute increase, bottle is filled with water but actual pressure upon the water comes from nitrogen gas.

Eight Kevlar 49/UF-3283 prepreg (First Stage MX) 5.75-inch diameter bottles were subjected to three different solvents to determine the effect of the solvents on the structural integrity of the case. The bottles were filled completely with solvent and allowed to stand at room temperature for twenty-four hours. After this soaking period, the bottles were drained and allowed to dry for 18 hours under vacuum at ambient temperature. The THF soaked bottles deteriorated between eight and twenty-four hours. With cyclohexane as solvent, the cases did not deteriorate like the THF soaked bottles; however, the insulation did pull away from the case walls. The ethyl acetate soaked bottles physically softened when observed after the soaking period and hardened again when dried. The insulation also pulled away from the case walls as did the cyclohexane but it did so to a greater extent. The results of the tests on the Kevlar bottles are summarized in Table XXVIII.

For an undetermined reason the first set of S-901 glass 5.75-inch bottles ruptured prematurely during hydrotesting. A new set of twelve 5.75-inch bottles was fabricated. Previously, 15 bottles were fabricated. Twelve were proofed to 80% of the control average, AVE,(three bottles). Four bottles burst during proofing, one bottle was cut in half to examine because of earlier failure and eight bottles were burst after proofing. Table XXIX summarizes the results of this first series of tests.

An additional set of twelve bottles was fabricated out of S-901/UF-3205 wet wind. These bottles had dome caps reinforcements. Three bottles were burst to establish the average pressure at failure and then 80% of this average pressure was calculated and the remaining nine bottles were hydro-proofed at this pressure. The bottles were filled (three per solvent) completely with solvent and allowed to stand at room temperature for 24

TABLE XXVIII. RESULTS OF HYDROTEST OF 5.75" KEVLAR BOTTLES (KEVLAR 49/FU-3283 PREPREG)

TWR-30684

Bottles Tested	Solvent	Exposure Conditions	Fiber Strength Average, KSI	CV %	Failure Mode	Comments
3			350	3.5	Polar	Control
3	Ethyl Acetate	24 Hrs @ Ambient	340	4.2	Polar	Only two bottles burst. Bottle #3 leaked at polar boss during test/no burst. This solvent affected the insulation the least.
3	THF	24 Hrs @ Ambient			Leaked at polar boss	Insulation separated from case during soaking period. Inspection after test showed swollen insulation. It appeared that the solvent migrated in between insulation and the Kevlar composite. Severe damage to composite around polar bosses.
2	Cyclohexane	24 Hrs @ Ambient			Leaked at polar boss	Solvent deteriorated the insulation. Some damage of composite was observed at polar boss.

NOTE: Some bottles soaked in THF and cyclohexane solvents had loose polar bosses prior to test.

TABLE XXIX. RESULTS OF HYDROTEST OF GLASS 5.75 INCH BOTTLES (S-901 GLASS/UF-3205 WET)   TWR-30684

Bottles Tested	Fiber Strength AVE, KSI	C.V. %	Failure Mode	Comments
3	421	7.7	Polar at dome, polor boss or knuckle region	Control
3	367	5.3		Burst after proofing 10/26/81
5	390	5.1		Burst after proofing 11/11/81
4*	-	-		Failed during proofing

* One bottle cut in half to examine cause of failure during proofing.

hours. After this soaking period, the bottles were drained and allowed to dry under vacuum for 18 hours at ambient. The results are shown in Table XXX.

It is concluded from the results of Tables XXVII through XXX that the extended use of solvents internally in the case, could cause severe damage to the case integrity, particularly to the case/insulation bond. These results indicate that special techniques would have to be employed when solvents are used extensively such as for propellant degradation to prohibit damage to the case resin system.

TABLE XXX.   RESULTS OF HYDROTEST OF 5.75" GLASS BOTTLES (S-901/UF-3205 WET)

Bottles Tested	Solvent	Exposure Conditions	Fiber Strength Average, KSI	CV %	Failure Mode	Comments
3			448	4.12	Polar cylinder	Control
3	Cyclohexane	24 Hrs @ Ambient	437	5.20	Polar at cylinder	This solvent had the least effect on the insulation. There was some swelling.
3	THF	24 Hrs @ Ambient			Leaked at cylinder	The solvent deteriorated the insulation completely leaving the composite exposed to the solvent. Due to crazing when hydroproofed, the THF solvent affected the resin matrix/fiber composite reinforcement.
3	Ethyl Acetate	24 Hrs @ Ambient			Leaked at cylinder	The insulation swelled and separated from case. Leaking of solvent through the polar bosses and in between insulation and case might have been the cause.

6.4.2 Solvent Effect on Fiber: Strength – Short Shear Beam Tests

The short shear beam test should measure the effect, if any, of the solvent upon the fiber strength. Two sets of tests were performed. In the first, NOL rings were made and cut into the small samples prior to the solvent exposure. It was then reasoned that the solvent migration into the cut ends may affect the results. A second set of four NOL rings were fabricated and the entire ring was subjected to solvent, then dried before being cut into the individual samples and tested.

The results presented below indicate that this test is extremely resin content sensitive. As the resin was affected by the solvent, the shear strength decreased significantly. Table XXXI shows the effect of various chemicals on the yarn and roving of Kevlar 49. This table indicates that only acids and strong bases significantly affect this fiber. The results of the short shear beam tests in Table XXXII show a very significant decrease in sample strength for the Kevlar with most solvents. It is concluded, therefore, that the resin system, UF-3283, used with the Kevlar fibers was degraded by the solvents. The resin system, UF-3205, used with the glass was degraded with only a few solvents.

TABLE XXXI.   Decrease in Strength of Kevlar 49 Due to   TWR-30684

Exposure to Various Chemicals

Source: Kevlar Data Book, Dupont

PROPERTY	VALUE	REF.
RESISTANCE TO CHEMICALS, ROOM TEMP. STRENGTH DECREASE IN 24 HOURS (EXCEPT WHERE NOTED)		II-11
CONCENTRATED ACIDS		
ACETIC (99.7%)	NONE	
BENZOIC (3%, 100°C, 100 HR)	26%	
FORMIC (90%, 100 HR)	7%	
HYDROCHLORIC (37%)	NONE	
HYDROFLUORIC (5%)	NONE	
(48%)	10%	
HYDROBROMIC (10%, 1000 HR)	60%	
NITRIC (1%, 100 HR)	5%	
(70%, 24 HR)	60%	
PHOSPHORIC (10%, 100 HR)	1%	
SALICYLIC (3%, 100°C, 1000 HR)	NONE	
SULFURIC (1%, 1000 HR)	5%	
(10%, 1000 HR)	31%	
(70%, 1000 HR)	59%	
(96%, 24 HR)	100%	
CONCENTRATED BASES		
AMMONIUM HYDROXIDE	NONE	
POTASSIUM HYDROXIDE	25%	
SODIUM HYDROXIDE	10%	

YARN AND ROVING OF KEVLAR® 49

CHEMICAL PROPERTIES

YARN AND ROVING OF KEVLAR® 49

CHEMICAL PROPERTIES

PROPERTY	VALUE	REF.
SOLVENTS		II-11
ACETONE	NONE	
BENZENE	NONE	
CARBON TETRACHLORIDE	NONE	
DIMETHYL FORMAMIDE (DMF)	NONE	
METHYLENE CHLORIDE	NONE	
METHYL ETHYL KETONE (MEK)	NONE	
TRICHLOROETHYLENE	1.5%	
TRICHLOROETHANE	NONE	
TOLUENE	NONE	
ALCOHOLS		II-11
BENZYL ALCOHOL	NONE	
ETHYL ALCOHOL	NONE	
METHYL ALCOHOL	<1%	
OTHER CHEMICALS		II-11
FORMALIN	1.5%	
"FREON" 11 (21 DAYS, 60°C)	2.7%	
"FREON" 22 (21 DAYS, 60°C)	3.6%	
GASOLINE	NONE	
JET FUEL	4.5%	
KEROSENE (21 DAYS, 60°C)	NONE	
OIL, LUBRICATING	NONE	
OIL, TRANSFORMER (21 DAYS, 60°C)	NONE	
WATER, SALT (NaCl SOLUTION)	<0.5%	
WATER, SEA (NEW JERSEY) (12 MONTHS)	1.5%	
WATER, BOILING (100 HRS)	2%	
WATER (TAP)	NONE	

THE EFFECT OF CHEMICALS ON THE
TENSILE PROPERTIES OF KEVLAR® 49 ARAMID

24-HOUR EXPOSURE

CHEMICAL	TENSILE STRENGTH (PSI)	TENSILE MODULUS (PSI x 10^{-6})
None (Control)	411,000	18.33
Acetic acid (99.7% CH_3COOH)	431,600	18.16
Formic acid (HCOOH)	361,900	17.99
Hydrochloric acid (37% HCl)	419,200	17.80
Nitric acid (70% HNO_3)	165,200	17.40
Sulfuric acid	Too weak to test.	
Ammonium hydroxide (28.5% NH_3)	423,800	17.91
Potassium hydroxide (50% Solution)	305,900	17.69
Sodium hydroxide (50% Solution)	369,500	17.45
Acetone	423,100	18.22
Benzene (C_6H_6)	420,900	17.91
Carbon tetrachloride (CCl_4)	422,000	18.46
Dimethylformamide (DMF)	418,600	17.97
Methylene chloride (CH_2Cl_2)	425,900	18.30
Methyl ethyl ketone (MEK)	424,600	17.98
Trichloroethylene ("Triclene")	404,700	18.17
Chlorothene (1,1,1-Trichloroethane)	418,600	18.32
Toluene ($C_6H_5CH_3$)	413,600	18.27
Benzyl alcohol ($C_6H_5CH_2OH$)	412,300	18.08
Ethyl alcohol (CH_5OH)	417,000	18.02
Methyl alcohol (methanol)	407,500	17.90
Formalin (HCHO)	405,500	17.87
Gasoline (Regular)	419,900	18.37
Jet fuel (Texaco "Abjet" K-40)	393,400	18.09
Lubricating oil ("Skydrol")	422,700	18.08
Salt water (5% Solution)	410,100	16.92
Tap water	417,200	18.27

Yarns were tested using air-actuated 4-C cord and yarn clamps on an Instron test machine, at 10" gage length with 3 turns per inch twist added, 10% per minute elongation, and at 55% R.H. and 72°F.

Conversion factor: MPa(mega-pascals) = lb/in^2 x 6.895 x 10^{-3}

TABLE XXXII.   Results of Short Shear Beam Tests on
Solvent-Exposed Composite Case Samples

| | Material Strength | | | |
| | Glass S901 | | Kevlar 49 | |
Solvent	$\overline{S}_H$	C_V	$\overline{S}_H$	C_V
Cyclohexane	9329 psi	1.4	4550 psi	2.0
Carbon Tetrachloride	8869 psi	2.0	1954 psi	11.0
Benzene	8314 psi	5.0	2005 psi	6.2
Carbon Disulfide	8575 psi	5.5	3669 psi	1.5
Toluene	8959 psi	5.2	1523 psi	7.6
M-xylene	8731 psi	2.3	977 psi	10.5
Chloroform	6713 psi	5.1	1336 psi	2.5
THF	8565 psi	6.3	950 psi	9.8
Methylene Chloride	4688 psi	1.2	2082 psi	2.0
Isopropanol	9006 psi	8.6	4538 psi	3.3
Methanol	8933 psi	1.5	2418 psi	7.0
Ethyl Acetate	8903 psi	.54	1457 psi	6.3
MEK	8942 psi	2.0	1732 psi	2.6
Acetone	8573 psi	5.5	1596 psi	1.5
Ethylene Glycol	9025 psi	1.1	4859 psi	1.4
Acetonitrile	8619 psi	2.2	1515 psi	16.0
DMSO	8927 psi	3.3	250 psi	8.3
S901/UF-3205 Control	9144 psi	6.0		
Kevlar/UF-383 Control			4841 psi	3.4

Definitions

$\overline{S}_H$ = Ultimate Stress = $\dfrac{0.75\ P_B}{bd}$ Where P_B = Pounds of Load

C_V = 100X $\dfrac{\text{Standard Deviation}}{\text{Mean Value}}$

b = Sample Width
d = Sample Thickness

Test:

ASTM D2344
Three tests per solvent (including control) = 108 tests

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The results for the first series of short shear beam tests are as follows:

NOL rings were prepared out of two material systems: S-901/UF-3205 wet wind and Kevlar/UF-3283 prepreg.

S-901/UF-3205 cure: $2\frac{1}{2}$ hours, 212°F, $2\frac{1}{2}$ hours 256°F, $2\frac{1}{2}$ hours 312°F.
Kevlar/UF-3283 cure: 8 hours 210°F

Specimens were machined in a 5:1 ratio of span to depth. Three samples were used per solvent and compared against control samples to determine if case materials were degraded by solvent exposure.

Sample Exposure

1. Specimens subjected to solvents for 24 hours totally immersed.
2. Temperature of solvent, ambient (approximately 70°F).
3. After immersion, samples were dried in a vacuum oven without heat for 24 hours.
4. Specimen was then sealed and labeled.

Sample Testing

1. Cross-head speed of load, .05"/minute
2. Chart speed, 2"/minute
3. Load range, 0-600 pounds
4. Temperature of test, 70°F.

Summation

For the S-901/UF-3205 system, based upon propellant solution and $\bar{S}_H$ (average shear strength) the solvents of the set that might be used to reclaim the case with lower risk are:

1. cyclohexane
2. ethyl acetate
3. MEK

For the Kevlar/UF-3283 system, based upon propellant solution and $\bar{S}_H$ (average shear strength) the solvents of the set that might be used to reclaim the case with lower rick are:

1. cyclohexane

Isopropanol and ethylene glycol do not degrade the material but do not appear to be candidates for propellant removal.

The results for the second series of short shear beam tests are summarized below:

Sample Preparation

NOL rings were fabricated from two material systems: Kevlar/UF3283 prepreg and S901 glass/UF3205 wet wind. Kevlar rings were cured for eight hours at 210 $\pm$ 10°F and glass rings for 2 1/2 hours at 256°F and 2 1/2 hours at 312°F.

NOL Rings Exposure

Four NOL rings were fabricated from each material system. Exposure conditions and observations are summarized in Table XXXIII.

Short Beam Sample Preparation

NOL rings subjected to solvents and control were cut into SBS in a 5:1 ratio of span to depth. Ten samples were cut from each NOL ring and submitted for testing.

Sample Testing

The samples shall be tested as follows:

    a. Cross-head speed, 0.05 in/min

    b. Chart speed, 2 in/min

    c. Load range, 0-600 lbs

    d. Test temperature, 70°F

The results, given in Table XXXIV, are in fair agreement with the results presented previously. They show that the glass system was unaffected by the three solvents tested whereas the Kevlar system was greatly affected by ethyl acetate and THF and not affected by cyclohexane.

TABLE XXXIII. NOL RINGS EXPOSURE

SOLVENT	S-901/UF-3205 NOL RING	KEVLAR/UF-3283 NOL RING	EXPOSURE CONDITIONS	DRYING CONDITIONS	OBSERVATIONS
THF	1	1	24 hours at amb	18 hours at amb under vac	When removed from THF and ethyl acetate solvents, the rings were pliable and appeared that the resin was softened. In the residue solvent, there appeared to be droplets of resin from the Kevlar rings. However, this effect was more noticeable with the rings soaked in THF.
Ethyl Acetate	1	1	→	→	
Cyclohexane	1	1			Cyclohexane solvent did not affect the rings as much as THF and ethyl acetate. Rings remained rigid when removed from the solvent. There was some resin residue from Kevlar rings but not to such an extent as with the THF and ethyl acetate rings.

TABLE XXXIV. RESULTS OF SHORT BEAM SHEARS SOLVENT EXPOSURE

MATERIAL SYSTEM	SOLVENT	EXPOSURE CONDITIONS	NUMBER OF *SAMPLES	SHEAR STRENGTH, psi	C.V. %	COMMENTS
Kevlar/ UF3283 Prepreg	------	------	10	5007	5.5	Control
Kevlar/ UF3283 Prepreg	Cyclohexane	Soaked for 24 hours at amb	10	5075	1.6	
Kevlar/ UF3283 Prepreg	Ethyl Acetate	Soaked for 24 hours at amb	10	1930	4.5	
Kevlar/ UF3283 Prepreg	THF	Soaked for 24 hours at amb	9	1476	2.5	
S-901/ UF3205 Wet	------	------	10	8908	2.9	Control
S-901/ UF3205 Wet	Cyclohexane	Soaked for 24 hours at amb	10	8972	3.6	
S-901/ UF3205 Wet	Ethyl Acetate	Soaked for 24 hours at amb	10	8818	1.7	
S-901/ UF3205 Wet	THF	Soaked for 24 hours at amb	10	9010	2.8	

*Cut from exposed NOL rings

6.4.3   Solvent Effect on Resin - RDS Tests

Rheometric Dynamic Speccroscopy (RDS)

Torsional stress is applied to a sample (2.5" x 0.5" x 1.8") rectangular in
shape. The frequency of torsion is held constant. Temperature is increased in
steps from 40°C to 180°C. Two values are reported for the glass transition
temperature. The temperature of which G" (the loss modulus) is a maximum and
the temperature at which tan delta is a maximum. Tan delta is the ratio of
G" /G' where G is the storage modulus. In theory, if the resin is plasticized
or improperly formulated, a lowering of the glass transition temperature is
observed as compared against a control sample. With solvent migration through
the case matrix, crosslinking may be decresed due to the breakdown of chemical
bonds and the solvent may become interspersed in between polymer chains causing
plasticization. Both of these occurrences would lower the glass transition
temperature as measured by this method relative to a control specimen. It should
be mentioned that this method is very resin dependent, ideally the fiber contributes
little unless the resin weight percent is very low, then the fiber becomes
significant.

RDS Samples

RDS samples were fabricated from prepreg Kevlar/UF 3285 (Ferro 6304-0031 Spool
#402) and S901 glass/UF 3205 wet wind. The Kevlar samples were cured for eight
hours at $210 \pm 10°F$ and the glass samples with C-4 cam (2 1/2 hours at 212°F,
2 1/2 hours at 256°F, 2 1/2 hours at 312°F).

Samples were cut to 1/2" wide x 2 1/2" long and 0.10" thick.

Samples were exposed to various solvents and dried prior to testing. Two samples
were used as control. The results obtained are summarized in Table XXXV.

The numbers under tgG are the glass transition temperatures and those under tgtanδ
is a ratio of $G"$/G' where G" is the loss modulus (or plastic) and G' is the elastic
modulus.

TABLE XXXV. RESULTS OF RDS TESTING OF THE EFFECT OF SOLVENT EXPOSURE OF THE CASE SAMPLES   TWR-30684

A. S901 Glass C̄ UF 3205

	TgG"	TgTanδ
B – Control	146	153.5
C – Control	147	155
A – Acetone	150	159
B – Cyclohexane	147	157
B – Ether	149	156.5
C – Ethyl Acetate	144.5	154.5
A – Methanol	150.5	156.5
C – Methylene Chloride	(158.5/111)*	164.5/114
THF	148	.159.5

*Non-standard shape of G" curve indicates slightly softened resin lost solvent to return to original properties

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TABLE XXXV (CONTINUED)

B.   RDS - Kevlar Results

6304-0031-402 Kevlar	TgG" °C	TgTan δ °C
K – Control	78	84
G – Control Qualification Run	79.5 80	85.5 88.5
H – Cyclohexane	80.5	86
H– Methylene Chloride	87.5	95.5
G – Ethyl Acetate	<40	63.5
G – Methanol	56.5	71.5
K – Ether	49	56
K – THF	49	62.5
G – Acetone	52	Test prematurely aborted due to mechanical problem

It is observed that the glass system was not affected by the various solvents tested whereas the Kevlar system was generally affected. These results are in good agreement to the results of the short shear beam tests.

6.5.1   Hydromining

Thirty impact tests were conducted on case samples, with and without insulation.
These tests confirm that hydromining is a questionable, high-risk method of
removing insulation even from glass cases. The most useful result is that
during low-pressure, hot-water hydromining of propellant, the insulation forms
a sufficient barrier to protect the case. The observations and test conditions
used are summarized in Table XXXVI.

Figures 25 through 28 show photographs of the insulation and case samples impacted
by the high pressure water. Figure 25 shows damage to EPDM rubber from an MX
case. Figure 26 shows the effect of the water on the V-45 (NBR) insulation from
a Third Stage Minuteman III case. Figure 27 shows the damage caused by impacting
the outside of an MX case (Kevlar system). Figure 28 shows the effect of the
water impact on the outside of a Minuteman III case with the external insulation
still intact.

It was concluded from the results of these tests that low pressure, hot water
hydromining could be conducted in a manner which would not damage the internal in-
sulation. Rate of travel and angle of impingement were both important parameters
which would govern the design of the equipment. It would be very important to
include an interlocking system which would shut off the water flow when the travel
stops. Any extended dwell in one place could damage the insulation.

TABLE XXXVI. HIGH PRESSURE WATER IMPACT TESTS ON CASE AND INSULATION

Case	Insulation	Nozzle Diameter, Inches	Test Number	Temperature °F	Pressure psi	Velocity Feet/Second	Sweep Cut Speed Inches/Second	Sweep Cut Damage	Dwell Cut Time, Seconds	Dwell Cut Damage	Impact Angle ⊥ or /45
Glass	None	.125	148	65	500	128	.8	Just thru cork	30	Jell Coat	⊥
Glass	None	.085	145	65	1000	255	.8	cork "	40		/45
Glass	None	.055	139	65	1800	276	.8	Cut Cork	30	Jell Coat	
			141	65	1800	276	.8	None	30	Jell Coat	
Glass	None	.055	135	65	3700	659	.8	Cut Cork	30	Jell Coat	
			137	65	3700	659	/9	"	30	Slight Case	
Glass	NBR	.125	142	65	500	128	.8	None	30	Slight	
			144	65	100	255	.8	None	30	Through Insulation	
Glass	NBR	.085	138	65	1800	276	.8	Slight	30	Jell Coat Through Insulation	
			140	65	1800	276	.8	None	30		
Glass	NBR	.055	134	65	3700	659	.8	Cut thru	30	Cut thru	
			136	65	3700	659	.8	Some	30	Cut thru	
Kevlar	None	.125	117	65	500	128	1.3	None	30	Yes	
			119	65	500	128	.8	None	30	Yes	
			120	65	500	128	.8	None	30	No	
			121	65	1000	255	.7	None	30	Severe	
			122	65	2000	393	.7	None	30	Severe	
Kevlar	None	.085	123	65	1500	276	.5	Slight	30	Penetration	
			125	65	1500	276	.8	Slight	30	Significant	
			128	170	10000	828	.7	½" Deep	2	Through	
Kevlar	None	.055	131	65	3700	659	.7	Slight	30	½" Deep	
			133	65	3700	659	.8	Slight	30	3/8" Deep	
Kevlar	EPDM	.125	116	65	500	128	.8	No	30	Yes	
			118	65	500	128	.8	No	30	Yes	
Kevlar	EPDM	.085	124	65	1800	276	.8	Slight	30	Slight	
			126	65	1800	276	.8	Slight	30	Significant	
			127	170	10000	828	.7	½" Deep	15	Through Delamination	
Kevlar	EPDM	.055	129	170	10000	1186	.8	½" Deep	5	Through Delamination	⊥
			130	65	3700	659	1.3	1" Deep	30	½" Deep Delamination	⊥
			132	65	3700	657	.8	Cut Thru	30	Significant	/45

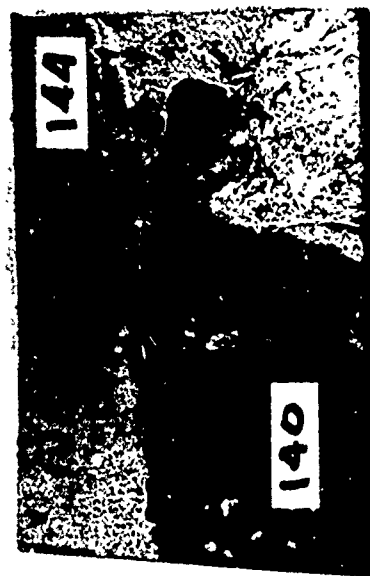FIGURE 25. SECTIONS OF MX CASE WITH EPDM INSULATION IMPACTED WITH WATER JET.

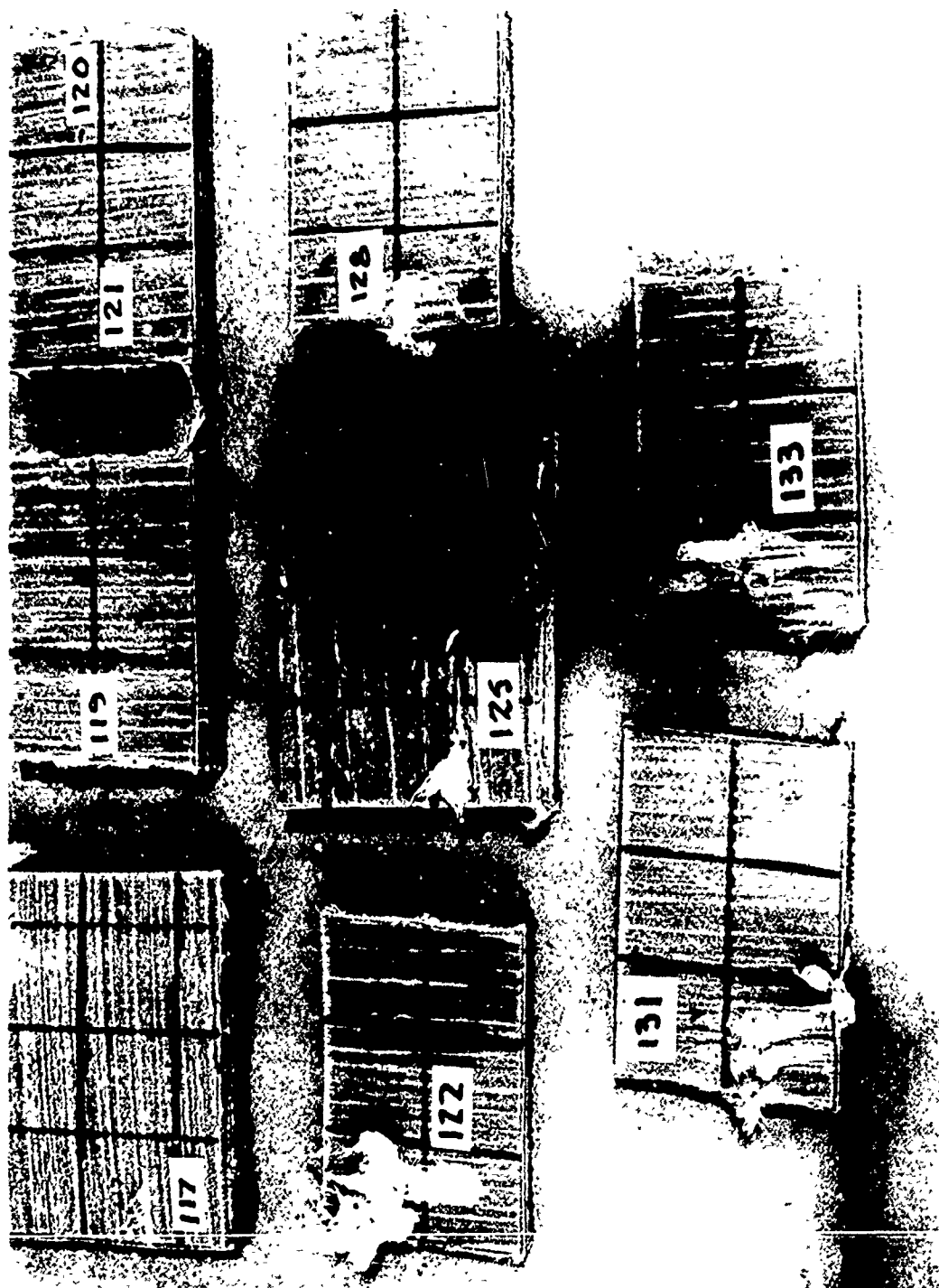FIGURE 26. SECTIONS OF MM III CASE WITH V45 INSULATION IMPACTED WITH WATER JET.

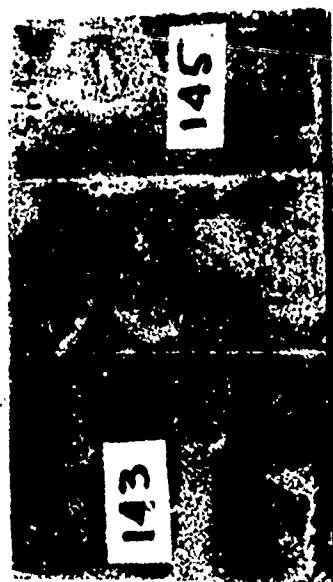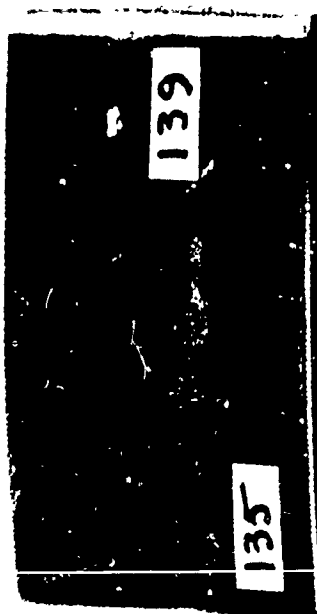FIGURE 27. SECTIONS OF MX KEVLAR CASE SHOWING DAMAGE DONE BY DIRECT IMPACT OF THE WATER JET.

FIGURE 28. SECTIONS OF MM III GLASS CASE SHOWING THE EFFECT OF DIRECT IMPACT OF THE WATER JET ON EXTERNAL INSULATION.

6.6   Reinsulation

It was concluded earlier that no attempt would be made to salvage the flaps
in any case salvage operation. Reinstallation of the flaps does not require
any new development since the replacement is the same operation as the original
installation. However, possible damage to the insulation during the propellant
or flap removal or the necessity to possibly remove part of the existing
insulation and bond new insulation to the remaining insulation required that
the possible effect of heat or solvent on the rebonding process be examined.

6.6.1   Effect of Heat on the Insulation

During the burnout method of propellant removal, the surface of the insulation
will be charred and the insulation will be subjected to varying degrees of
heating. The objective of these tests is to determine whether the burnout
process will affect the integrity of the rebuilt case: (1) Can virgin rubber
be bonded or vulcanized to the remaining insulation and (2) will the bond
between the insulation and flaps be equal to the original installation.

For this evaluation, samples of fired motors, MX first stage and Minuteman
III third stage was obtained. The char was then removed, the exposed
insulation is then prepared and virgin insulation was reapplied and cured to
the old insulation. 180° peel tests and tensile adhesion tests will confirm
the effectiveness of the reinsulation process.

Sample Preparation. When possible samples of insulation were obtained from
sections of fired cases. The char was then removed down to uncharred insul-
ation, buffed to a uniform surface, cleaned with a light solvent wipe, painted
with adhesive and dried then virgin insulation was applied and cured to the old
rubber.

When uncured V45 insulation was bonded to original case-bonded insulation of a section of a fired case, the post vulcanized bond between the fired V45 and new V45 was greater than the bond between the insulation and the case. Failure always occurred at this interface and no quantitative data were obtained. In order to obtain quantitative results the insulation was removed by peeling the insulation from the case, aided by a small quantity of MEK solvent. The fired insulation was then bonded with Chemlock 205-233 and dioctyl phthalate (DOP) to steel peel coupons. Uncured V45 was then bonded as before to the fired V45 insulation. The results show that the insulation to insulation bonding produced a very good PVC bond. All failures were cohesive but appeared to be in the ply bond of the new uncured rubber.

A similar procedure was employed for removing EPDM 053A insulation from a section of a tested MX case (DM-1). When DOP was used as the PVC activator, the peel values were similar to or slightly higher than for the control and appeared adhesive. The fired insulation to steel bond remained intact. Results of the above tests are summarized in Table XXXVII.

6.6.2   Effect of Solvent on Reinstallation of Insulation and/or Flaps

In the event that solvents are used to remove the propellant and/or liner, the insulation also would be subjected to the solvent. The objective of these tests is to confirm that the reinstallation of the flaps or rebonding new insulation to repair damage would not be adversely affected by the solvents.

Samples of EPDM and V45 insulation are subjected to a solvent soak. The samples are then dried by vacuum drying and 180° peel samples and tensile adhesion samples are prepared by bonding cured rubber to cured rubber. -

To evaluate the effect of the various solvents, a new test procedure was developed to isolate the effect in the rubber to rubber bond.

TABLE XXXVII. EFFECT OF HEAT ON INSULATION – 180° PEEL TESTS[1]   TWR-30684

A. Virgin 053A to Fired 053A

Construction	PVC Activator[2]	High	Average	Low	Failure	Samples
Steel/Chemlok 205-236/ Fired 053A/Chemlok 236/ Uncured virgin 053A	None	70.5[4]	64.0	45.0		1
Same as A.	DOP	78.0	71.0	55.0	Separation from steel	1
Same as A.	parafin₁ oil [3]	67.0	48.0	33.5	Separation from steel	1
Same as A.	None	47.7	43.5	37.4	All adhesive failures	5
		Initial	Maximum	Average		
Steel/FM123[5] Fired 053A/Chemlok 205-236 Uncured 053A	None	32.2	34.6	13.4	Adhesive – failed in Chemlok adhesive	3
Steel/FM123/Fired 053A/ Chemlok 205-238/ Uncured 053A	None	37.3	41.2	15.1	Adhesive – failed in Chemlok	3
Steel/FM123/Fired 053A/ FM123/Cured 053A	None	12.0	23.7	16.9	Adhesive – failed in FM123	3
Steel/FM123/Fired 053A/ ER2216/Cured 053A	None	16.9	16.9	4.2	Adhesive – failed in Epoxy Resin	3

Notes:

(1) All 180° peel tests were pulled at 10"/minute, and room temperature.
(2) 10% solution of PVC adhesion activator applied to dried Chemlok adhesive
(3) Cyclolube 85
(4) All peel test results in pli units.
(5) Epoxy film adhesive supplied by American Cyanamid.

B. Virgin V45 to Fired V45

Construction		High	Average	Low	Failure	Samples
Steel[2]/Epoxy FRP Case/ Fired/Uncured Virgin		177[4]			100% Cohesive	
Steel[2]/Chemlok 220-233- DOP[1]/Fired/Uncured Virgin		116	107	98.2	100% Cohesive	All samples had plylock separation.

Notes:

(1) A 10% solution of dioctyl phthalate was applied to the Chemlok coated surface to activate post vulcanized cure (PVC) adhesion of the fired insulation to the steel peel coupon.
(2) Steel peel coupons were used to increase the modulus of the fired insulation of the FRP case material.
(3) 180° peel tests were pulled at 10"/Minute and room temperature.
(4) All peel test results in pli units.

The new tool consisted of a steel mandrel 9" long by 6" wide and 3/4" thick. The mandrel is overwrapped with one of the case constructions with the inside case wall out. The case/mandrel assembly is cured with the respective precured insulation. These samples are then treated with solvent. 180° peel testing is performed at this time and also after additional precured insulation has been post vulcanize bonded. The latter is to represent the rebonding of a precured flap after solvent removal of the propellant. A sketch of the overwrap mandrel is shown in Figure 29. The procedure for making the test specimen is given in Table XXXVIII.

One set of overwrap mandrel specimens was wound and cured with the MX 053A EPDM insulation. The mandrels were cut into individual specimens and drilled and threaded so that they could be mounted in the Instron for testing. Before testing the mandrel, specimens were contacted with solvent for 24 hours then dried. The insulation layer was then cut or slit into 5-1" wide 150-180° peels.

Samples were also made using V45 rubber and glass fiber and resin system similar to that used on the Minuteman III third stage cases. The results, giving 180° peel data for the bond between the insulation and the case, are summarized in Table XXXIX. The mechanical properties of the rubbers used in the above tests are given in Table XXXX.

It was concluded from the above tests that cyclohexane, tetrahydrofuran (THF) and chloroform were the most detrimental solvents on the EPDM 053A insulation. Solvents having the least effect on EPDM were acetone, isopropanol and ethylene glycol. Methyl ethyl ketone (MEK), THF, ethylene dichloride and dimethyl foran amide (DMF) were the most detrimental to the V45 (NBR/Hi Sil 215) insulation. Solvents having the least effect on V45 were cyclohexane, ethylene glycol and isopropanol.
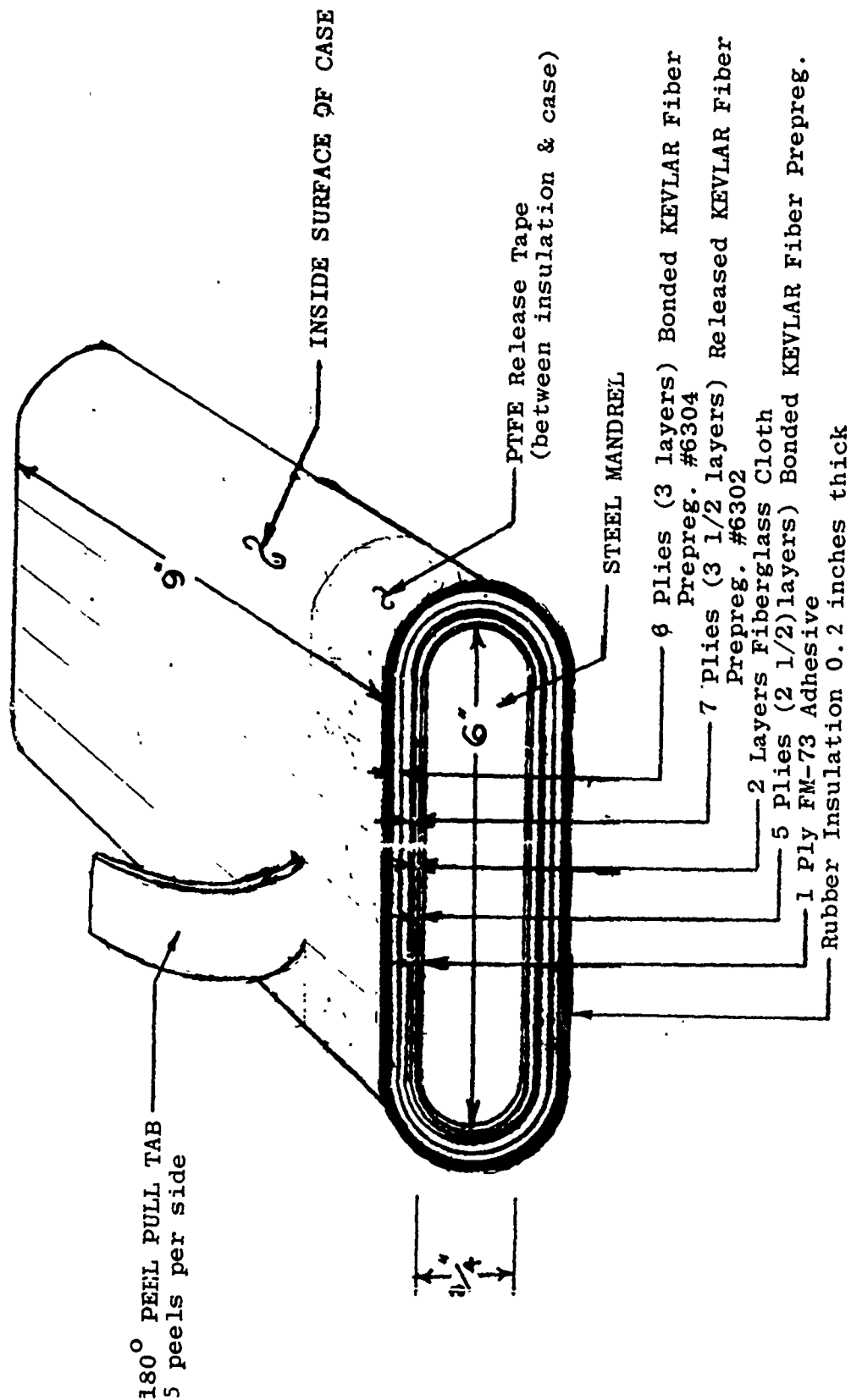
FIGURE 29. SKETCH OF MANDREL FOR PREPARATION OF OVER WRAP MANDREL SPECIMEN

INSIDE SURFACE OF CASE

PTFE Release Tape
(between insulation & case)

STEEL MANDREL

8 Plies (3 layers) Bonded KEVLAR Fiber
Prepreg. #6304

7 Plies (3 1/2 layers) Released KEVLAR Fiber
Prepreg. #6302

2 Layers Fiberglass Cloth

5 Plies (2 1/2) layers) Bonded KEVLAR Fiber Prepreg.

1 Ply FM-73 Adhesive

Rubber Insulation 0.2 inches thick

180° PEEL PULL TAB
5 peels per side

TABLE XXXVIII. PROCEDURE FOR PREPARATION OF OVERWRAP SAMPLES   TWR-30684

A. **MX Simulated Case Overwrap[1] Mandrel Construction**

Materials of Construction

Roving:	Kevlar/UF-3283 prepreg Kevlar - DC20[2] coated/UF-3283 prepreg
Glass Cloth:	341 glass cloth[3]/UF-3283
Film Adhesive:	FM73[4]
Steel Mandrels:	Grit blasted and degreased
Insulation:	053A EPDM/CR/HiSil 233 rubber insulation, 2 precured pads 5½" x 8" x 0.2", insulation cure was 300°F x 120 minutes x 100 psig CO_2; bonding surface of cured insulation was lightly abraded and cleared with MEK solvent; 3" wide PTFE tape was applied for peel tab release at the rubber/FRP interface.
Winding Detail/Winding Machine:	McClean-Arderson - small Building M-8
Hoop Winding Gear Ratios:	120/20, 120/20, 56/56, 28/84, 113/36
Tension:	10 pounds
Elevational Plan:	x = ply

                                 Steel mandrel
                                 1 x FM73
                                 5 hoop x Kevlar/UF-3283
                                 2 x 341 glass cloth/UF-3283
                                 7 hoop x Kevlar - DC20/UF-3283
                                 6 hoop x Kevlar/UF-3283
                                 1 x FM73

Formulation of UF-3283:		%
	Shell EPON 828	40.10
	Shell EPON 871	20.05
	Cibas Geigy Araldite 906	39.30
	EMI-24	0.55
Cure:	Vacuum bag Cured in oven @ 210°F for 8 hours	

Notes:

(1) See Figure 29 for overlay mandrel
(2) Dow Corning 20 release agent
(3) Woven glass cloth
(4) Epoxy film adhesive supplied by American Cyanamide

TABLE XXXVIII (CONTINUED)

B. <u>Minuteman III Case Simulation Overwrap Mandrel Construction</u>

Materials of Construction

Roving:	S-901 Glass – 20 end
Resin:	UF-3205
Steel Mandrels:	Grit blasted and degreased
Insulation:	NBR/HiSil 233 rubber insulation; 2 cured pads 5½" x 8" x 0.2"; insulation was cured at 300°F x 150 minutes x 100 psig CO_2; bonding of cured insulation was lightly abraded and cleaned with MEK solvent; 3½ wide PTFE tape was applied for peel tab release at rubber/FRP interface.
Winding Detail/ Winding Machine:	Small McClean-Anderson Building M-8 Resin, UF-3205 was hand applied during the winding operation
Hoop Winding Gear Ratios:	120/20, 120/20, 56/56, 28/84, 113/36
Tension:	5 pounds
Elevation Plan:	(x = ply)

Steel Mandrel
Gel coat UF-3205
5 hoop x S-901 glass roving (roving is thoroughly wet out
  with resin)
2 x glass cloth predipped in UF-3205
5 hoop x S-901 glass roving/UF-3205

Formulation of UF-3205:

	%
Ciba-Geigy Araldite 6005	52.28
Nadic methyl anhydride	47.04
Benzyl Dimethylamine	0.68

Cure: Vacuum bag
Cured[1] in oven @ 212°F for 2½ hours, 256°F for 2½ hours,
and 312°F for 2½ hours.

Note:

(1) Trident C4 cam cure; all temperatures were additive.

TABLE XXXIX. RESULTS OF TESTS ON THE EFFECT OF SOLVENTS ON THE RUBBER-RUBBER BOND STRENGTH

A. MX Simulated Case Overwrap* 180° Peel Tests[1]

Solvent Treatment[2]	Time in Solvent Contact Units	High[3] pli	Average[4] pli	Low[5] pli	Failure Mode
Control	--	4.8	3.5	2.6	Adhesive
Ethyl Acetate	24 Hours	4.1	3.0	2.6	Adhesive
Ethyl Acetate	72 Hours	12.5	9.6	7.6	Adhesive
Methyl Ethyl Ketone	24 Hours	5.0	3.1	2.0	Adhesive
Cyclohexane	24 Hours	9.4	6.4	4.2	Adhesive
Cyclohexane	72 Hours	3.6	1.3	0.9	Adhesive
Ethylene Glycol	24 Hours	6.6	4.1	2.8	Adhesive
Tetrahydrofuran	24 Hours	3.7	2.7	2.0	Adhesive
Dimethyl Sulfone	24 Hours	7.2	5.6	4.3	Adhesive
Acetone	24 Hours	12.1	8.6	6.2	Adhesive
Isopropylalcohol	24 Hours	11.8	8.6	5.9	Adhesive
Chloroform	24 Hours	3.8	1.3	0.8	Adhesive
Dimethylformamide	24 Hours	12.8	9.7	7.3	Adhesive

Notes:
(1) Pulled at 10"/minute and ambient (75°F) temperature.
(2) Insulation was in direct contact with liquid solvent at ambient (75°) temperature and atmospheric pressure. Samples were dried in vacuum oven after solvent contact for 24 hours at ambient temperature.
(3) Initial fracture peak.
(4) Arithmetic average between highest and lowest points in failure profile.
(5) Lowest point in failure profile.
(6) Each point separates five tests.

* Construction: Elevational - FM73 film adhesive steel mandrel, Kevlar/UF-3283 Epoxy Resin Prepreg; 341 glass cloth/UF-3283; and Kevlar coated with DC20 silicone/UF-3283 prepreg; FM73 adhesive; 0.2" thick 053A rubber insulation. (See Figure 29)

B. Minuteman III Simulated Case Overwrap* - 180° Peel Tests

Solvent Treatment	Time In Solvent Contact Units	High pli	Average pli	Low pli	Failure Mode	Shore A Hardness Treated Side[2]	Untreated Side	Swelling[3]
Control	24 Hours	39.1	33.1	24.5		71	71	0
Ethyl Acetate	24 Hours	19.8	15.9	13.8		52	73	3
Methyl Ethyl Ketone	24 Hours	21.5	15.0	10.5		47	73	8
Methyl Ethyl Ketone	72 Hours	9.7	4.6	2.4		51	75	8
Cyclohexane	24 Hours	43.5	38.3	27.6		75	73	0
Ethylene Glycol	24 Hours	20.8	18.0	14.2		78	73	
Tetrahydrofuan	24 Hours	9.6	5.6	3.2		44	73	5
Dimethyl Sulfone	24 Hours	20.6	16.0	12.0		53	73	3
Acetone	24 Hours	32.1	28.6	24.8		49	77	10
Isopropyl Alcohol	24 Hours	21.1	16.9	12.4		71	71	
Chloroform	24 Hours	17.7	11.9	8.3		44	71	9
Dimethylformamide	24 Hours	11.4	6.8	4.0		47	66	

Notes:

(1) Average of five tests, instantainious, taken at ambient room temperature after solvent was vacuum dried from insulation.
(2) Area of insulation in contact with liquid solvent.
(3) Observable change in surface of insulation after vacuum drying; 10 is worst condition.
(4) Each point represents five tests.

*Construction: Elevation - steel mandrel; S-901 glass roving impregnated with UF-3205 Epoxy Resin; 0.2 V45 NBR - silica reinforced insulation

TABLE XXXX. TENSILE ADHESION TESTS -- SOLVENT IMPACT ON MECHANICAL PROPERTIES OF RUBBER INSULATION

	Units	Co	Ethyl Ether	Cyclo Hexane	DMF	Ethyl Acetate	Acetone	Methyl Alcohol	Methylene Chloride	DMSO	THF	ASTM Method
053A Stock: EPDM/CR-FB/HiSil 233 (Virgin) Cure: 300°F x 150' in press MTE: Pulled @ 20"/minute; 75°F; parallel												
Tensile	psi	2017	2066	1783	1919	1914	1860	1873	2021	1988	2136	ASTM D412
Elongation	%	675	663	646	688	642	601	746	726	726	752	
Modulus @ yield	psi	299	312	248	271	298	266	251	266	274	284	
Hardness – Shore A	inst/15"	64/57	70/62	67/55	62/54	70/58	67/56	66/52	68/60	66/55	64/52	ASTM D2240
V45 from Minuteman III Case Stock: NBR/HiSil 233 (Fired) Cure: Production cycle												
MECHANICAL PROPERTIES												
Tensile	psi	2325	2193	2003	1485	2105	2155	2070	2090	1585	2238	ASTM D412
Elongation	%	600	560	590	510	540	510	550	510	551	540	
Modulus @ yield	psi	398	397	330	305	400	410	378	418	299	407	
Shore A Hardness												ASTM D2240
Smooth	inst/15"	67/56	73/52	57/48	43/37	65/50	69/56	66/50	66/46	49/40	59/50	
Rough	inst/15"	66/54	71/55	56/44	42/36	64/48	64/52	61/50	63/50	49/39	60/51	
V45 Control Stock: NVR/HiSil 233 (Virgin) Cure: 300°F x 150' x 100 psig CO₂ + process case cure as AGC 36512												
MECHANICAL PROPERTIES												
Tensile	psi	2733	2785	2308	2578	2776	2734	2684	2956	2476	2780	
Elongation	%	591	576	584	595	596	603	623	595	637	600	
Modulus @ yield	psi	452	473	395	427	459	455	431	498	362	454	
Hardness – Shore A	inst/15"	69/50	74/59	59/45	63/50	75/58	74/58	72/55	76/58	56/41	72/58	

Notes:

(1) Solvent efficiency
a) 053: cyclohexane most efficient
    least efficient: ethyl ether, methylene chloride and THF

b) V45: Most efficient: DMF and DMSO
    Note that a large negative change in hardness has occurred. Both solvents have high boiling point and may not have been completely stripped off. Solvent residues may be acting as plasticizers or could have altered cure rheology.

(2) The increased hardness of the ethyl ether extracted V45 sample may be due to removal of the DOP plasticizer.

The results in Table XXXIX indicate the effect of solvent on rubber-rubber bond strength varies greatly from solvent to solvent. Some increase the bond strength and some weaken the bond. A significant decrease in the bond strength would be a factor in determining which solvent might be used for propellant removal.

6.7   Engineering Assessment of Case Salvage

An assessment of the acceptability of salvaged composite motor cases

for reusability indicates that there are no over-riding considerations

that would prevent reuse. This assessment considered:

a) The effects of propellant removal techniques on the case materials

and structure.

b) Refurbishment of the salvages case.

c) Re-proofing of the refurbished case in preparation for re-loading.

In addition, this assessment assumed:

a) That physical damage, such as cut fibers, would be treated in the

same manner as it would with a single use case (i.e. damage repair is not

unique to case salvage).

b) The case materials have not naturally aged to the point that they

could not meet a second service life requirement. Since composites are

commonly used in aircraft in far more severe environments than rocket

motors are typically exposed to, it is unlikely that a composite case would

naturally age beyond use.

Since this study was directed toward composite cases in general, and

not toward a specific motor, both glass/epoxy and Kevlar/epoxy were con-

sidered. Graphite/epoxy was not included, since there are no operational

motors utilizing graphite cases in service at this time. However, Thiokol

is currently assessing the salvage and reuse of graphite cases in support

of a feasibility study for a filament wound composite Space Shuttle Solid

Rocket Motor, and the results of that study were recognized in the case

salvage study.

The initial step in the case salvage process would be propellant

removal. The effects of three removal techniques on the case structure

were considered:

a) hydromining,

b) wet machining, and

c) solvent softening and removal.

Both hydromining and wet machining would subject the interior of the case to prolonged moisture. In addition, hydromining would require hot water, which could result in interior temperatures of up to 170°F. Both moisture and temperature are known to adversely affect the resin-dominated properties (primarily transverse and shear strength and modulus) of all composites, but these effects are completely reversible upon drying and cooling. Neither of these salvage techniques would adversely affect the case, since the insulator would prevent direct exposure of the composite to moisture and would attenuate the internal temperature. The moisture exposure would thus be similar to that experienced during normal post-manufacturing hydrotest. Normal post process drying would remove accumulated moisture.

The effects of solvents used to soften and remove propellant were also considered. A variety of laboratory scale composite specimens were exposed to various solvents and tested to determine their effects on individual material properties. The test results are discussed in detail in Section 6.4. The implications of these effects on case structural performance is discussed in this section.

The laboratory tests showed that the primary effect of the solvents was to degrade some resins, as evidenced by the degradation in interlaminar shear strength, and to degrade the rubber insulation, as evidenced by the degradation in the bond between the insulator material and the composite, and by the degradation of the pressure vessel bladders. The effect of solvents on fiber tensile strength was not really evaluated, since the degradation of the pressure vessel bladders precluded hydroburst testing. However, the effects of various chemicals on glass, Kevlar, and graphite have been

evaluated by their manufacturers and by other researchers, with indications that all three fibers are impervious to all but strong acids. The fiber tensile strength is therefore unlikely to be affected by any of the solvents that could be used for propellant removal.

The implications of these effects on the case structural performance depends on the specific design requirements for the motor case in question, since some properties are more significant to some cases than to others. Table XXXXI shows the design requirements typically considered in case design, and the corresponding significant material property.

A comparison of the case property requirements indicates that the use of solvents would not affect the case burst strength, since the fiber tensile strength would probably not be affected. However, this ignores the fact that all of the solvents severely degraded the insulator, which may in turn result in leakage and an inability to withstand pressure.

Stiffness and buckling strength are mostly influenced by the fiber modulus and, like tensile strength, would not be directly affected by solvent exposure.

The skirt and case external load capability are primarily a function of the composite compressive strength and the skirt-to-shear ply-to-case bond at the Y-joint. The compressive strength is a "fiber dominated" property, but is actually highly dependent on the resin due to the nature of compressive failure in composite materials, and degradation of the resin, as evidenced by either visual appearance or loss of interlaminar shear strength, strongly suggests a corresponding loss of compressive strength. Whether or not a given case would suffer degradation, and whether or not such degradation would be acceptable, would depend on the solvent used, the specific case material system, and the case strength requirements. The test results indicate that the material degradation depends on both the fiber/

TABLE XXXXI. COMPOSITE CASE DESIGN REQUIREMENTS AND MATERIAL PROPERTY COMPARISON

DESIGN REQUIREMENT	SIGNIFICANT MATERIAL PROPERTY
internal pressure	fiber tensile strength
stiffness	fiber modulus
buckling	fiber modulus
skirt & case external loads	composite compressive strength
	composite/elastomer bond strength
aeroheating	composite compressive strength
	composite shear strength
	resin glass transition temperature (T_g)

resin combination and the resin used. In addition, some cases have very low strength requirements, and thus could tolerate more degradation than others. Furthermore, these tests were based on specimens soaked in solvent. Presumably, the propellant removal process would be conducted in a manner that would preclude prolonged case contact with the solvent, although solvent could migrate through a damaged insulator and attack the composite.

Following propellant removal, the case may require refurbishment prior to re-loading. The only refurbishment step that could affect the case structure would be removal and replacement of the insulator (either partially or entirely), if this were necessary. Insulator removal would most likely be accomplished by locally heating the case/insulator bond directly with a hand-held heat gun, while applying a 90°-180° peeling load. The primary risk to the composite would be local fiber damage. However, our experience with peeling insulation from the Kevlar/epoxy MX first stage case has not shown any evidence of fiber damage. Since Kevlar is the fiber most prone to damage and fraying, this indicates that peeling the insulator will not adversely affect the case. The local heating would not have an effect since the temperatures are expected to be below 200°F, which is below normal case cure temperature. Local hot spots due to incorrect use of the heat guns could cause very local softening of the resin, but these would re-solidify and return to their initial state with no adverse effect on the case as a whole. The fibers would not be affected since all of them can tolerate several hundred degrees without degradation.

Replacement of the insulator, either for the entire case, if the insulator is totally replaced, or for a local area, if only a section of the insulator is replaced, would require an insulator bond line cure cycle. Neither of these would adversely affect the case, since the temperature is below the case cure temperature.

Following refurbishment, a hydroproof test will be required prior to propellant loading. The hydrotest may be limited to a low pressure leak test or it may entail a full hydroproof to 1.0-1.1 times MEOP. The decision as to which test to conduct will probably depend on the specific motor program requirements. A low pressure leak test would be of no concern, but a second full-proof cycle would be a significant departure from the customary single proof cycle. The effects of multiple proof cycles have not been extensively studied. However, a review of limited studies and testing by Thiokol and other researchers indicates that a second proof cycle will be tolerable. Subscale pressure vessel tests with Kevlar 49/UF-3283 in support of the MX Stage I program indicated that a single hydroproof cycle to below 85% of the actual burst pressure did not degrade strength (multiple proofing was not evaluated during these tests). Multiple cycling was studied in support of the composite Shuttle case study. T-300 graphite/UF-3283 pressure vessels were cycled to 72% of burst up to 40 times with no degradation in subsequent burst pressure.

Since proof tests are generally conducted at 70-75% of average burst, these tests, in conjunction with the high fatigue resistance typical of fiber-reinforced composites, suggest that a second proof cycle would not be detrimental. However, additional subscale hydroburst testing is recommended to fully define a) the proof level at which subsequent degradation in burst strength results, and b) the effects of multi-proofing.

INTER-OFFICE MEMO

9205-81-M080

TO:	K. B. Reynolds, Technical Services
CC:	J. W. Loosle, J. E. Engle, M. H. Phillips, W. L. Merrill, E. D. Brown, M. L. Levinthal
FROM:	D. W. Kase, Safety Analysis
SUBJECT:	Hazards Analysis No. 379 Propellant Machining With RDS-394 Cutting Tool

This study is a theoretical evaluation of the thermal hazard of machining propellants with the RDS-394 cutting tool. It is based on an analytical model developed by E. T. Hikida, Hercules Bacchus, as published in his paper "Analysis of Heat Generation from Dry Machining of Solid Propellant," 7 August 1972.

Input parameters given by you are as follows:

    Speed - to 1300 rpm
    Diameter - 5 in. max
    Feed - to 15 in/min

: parameters that determine the energy released upon propellant cutting are the cutter tip speed and the shear strength of the propellant. As derived by Hikida, in basic engineering units:

    q = 667 SV where S = shear strength - psi
                       V = tip velocity - ft/sec.
                       q = heat flux - Btu/ft²-hr.

Based on your parameters:

$$V = \pi DN = \frac{\pi \times 5 \text{ in} \times 1300/min}{60 \text{ sec/min} \times 12 \text{ in/ft}} = 28.36 \text{ ft/sec.}$$

It can be seen from the sketches below, that a worst case in terms of heat retention is zero feed.

zero feed   zero feed
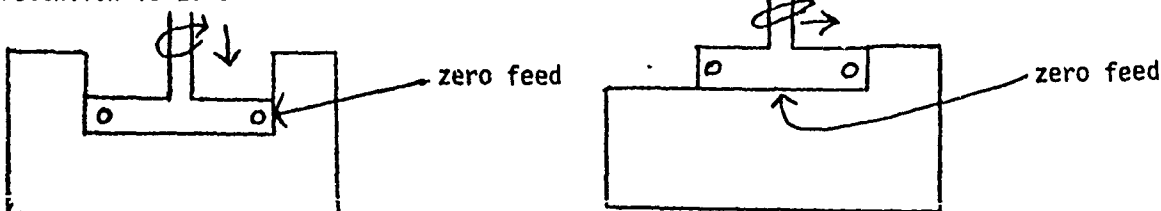
Therefore, in terms of heat dissipation, no credit can be given to chip removal .n traverse, but can be in the direction of the cut.

Thiokol / **WASATCH DIVISION**

Brigham City, Utah

heat generated then, is, for a 100 psi shear strength:

$$q = 667 \times 100 \times 28.36 - 1.892 \times 10^6 \text{ Btu/ft.}^2 - hr.$$

Tougher propellants will generate more heat, while slower speeds or weaker propellants will develop less. The key question is where does the heat go. As long as propellant is being cut and removed, its exposure to the flux is very short. However, the tool is exposed to the flux continuously as it cuts. And in an area of zero feed, hence no propellant removal, the calculation is unnecessarily conservative and severe. This is because the propellant is not being sheared, but rather only rubbed by the tool at some lower, but indeterminate interfacial friction pressure F. However, while S is replaced by a lower value F, the cumulative effect of successive passages of the tool surface must be allowed.

In this treatment, admittedly conservative, it is assumed that the heat generated is all absorbed in the item considered, that is, propellant chip, tool, or rubbed propellant surface.

1. **Propellant Chip Heating:**

A flux of 1.892×10^6 Btu/ft.^{2} - hr. can be related to any data which correlates the time-to-ignition with a flux exposure. Arc image exposure is a common method of flux exposure control, and data was provided for certain cross-linked double base (XLDB) propellants in the previously referenced report. By extrapolation to the present flux, exposures of 0.002 to 0.05 seconds will achieve ignition. nilarly, by extrapolation, data by Atwood et al* gives apparent ignition threshholds in the 2-10 msec range for VTG-5A propellant, at 1.892×10^6 Btu/ft.^{2}- hr. (142 cal/cm^2 - sec).

Derr and Fleming published data for composite propellants, "A Correlation of Solid Propellant Arc-Image Ignition Data", Lockheed Propulsion Co., Redlands, Calif., in which threshholds in the 10-20 msec range were measured.

So the question is, how long does a propellant element see this flux? The answer is, it sees it as long as it takes for the tool to pass. If one assumes that the entire cutter width (0.5 in) is generating flux, the time of exposure to its passage is given by:

$$t = \frac{1}{V} = \frac{0.5 \text{ in.}}{28.36 \text{ ft/sec.} \times 12 \text{ in/ft.}} = 0.00147 \text{ sec.}$$

In theory, a perfect knife edge passes in an infinitesimal interval. A contact surface of 0.05 in, will give a passage interval of 0.15 msec, while the full half inch gives an interval of 1.5 msec, approximating the threshholds published for XLDB. It would appear that given the sharp, relieved cutting edges described in RDS-394, the exposure time is sufficiently low to not constitute an ignition source. Note that a harder, tougher propellant, greater than 100 psi shear strength, will create higher fluxes, and if far different, should be reanalysed as above.

The effect of Aging on The Ignition of Trident VTG-5A Propellant, Atwood, Zurn Boggs, Price and Stayton, Naval Weapons Center, China Lake, Calif.

.. **Tool Tip Heating:**

Because the tool steel is a far greater absorber and conductor of thermal energy, it should be expected that most of the flux generated will be taken up by the tool. Hikida (op. cit.) calcualted, via a proprietary computer model, tip temperatures at 3 and 5 ft/sec. tip speed. In order to evaluate a tip speed of 28 ft/sec., it will be necessary to ascertain whether Thiokol/Wasatch Division Engineering can perform such a thermal modeling. The importance of tip temperature, theoretical or actual, is the effect on propellant if and when the tool stalls, fails and/or dwells on a fixed element of propellant. We have already seen that, while in steady state rotation, the hazard is acceptable. The return of heat from the tool to the propellant is necessarily only a part of the whole of the heat generated, and can not exceed the 100% assumed in Section 1 preceding, as long as rotation continues; i.e., exposure time remains less than 1 - 2 msec.

At even 5 ft/sec. tip speeds, the theoretical tool temperature approaches hazardous levels. The equilibrium temperature at the tip becomes a function of conductive path geometry, heat sink or dissipation capacity, the effects of convective cooling, and the actual distribution of generated heat between tool and chip. Actual measurements, using "Telatemp" dots, on the FD-0014 inert motor (ref. Hazards Analysis No. 2B, D. W. Kase to R. D. Hutchison, 17 July 1975) showed a maximum tool temperature rise of 29° F at about 1.1 ft/sec. in about 3 minutes. Extrapolation of these results to 28 ft/sec. and any greater machining times would be exceedingly tenuous, and not considered valid.

you can see, tool temperature analysis is a nearly un-analysable conundrum. The solution in large scale machining operations, where the consequences of ignition would be catastrophic, has been and continues to be, water flooding. Any error or uncertainty in analysis, even the cutting into a sub-surface chunk of metal, and (as has happened) the fracture failure of the cutting tool, if forgiven by the overwhelming heat sink of the water.

Dry machining as a case salvage approach can only be considered safe, at our present state of knowledge, if (1) there are no foreign objects in the propellant, (2) the tool cannot fracture, and (3) it cannot stall or dwell.

3. **Rubbing Contact (No Feed):**

As stated earlier, the fundamental question is, what interfacial friction pressure obtains at zero feed? It is less than the shear strength, and, if contact is maintained, greater than zero. An obviously conservative approach is to assume the flux is as determined previously for shear-cutting, and evaluate the cumulative effect of successive blade passages. The question then is whether the surface, heated by the blade passage, will return to its original temperature before the next passage, or if not, what residual ΔT will remain.
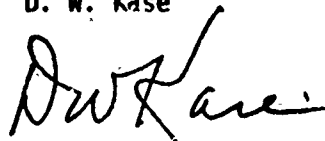
The interval between blade passages is:

$$t_1 = \frac{60 \text{ sec/min.}}{1300/\text{min} \times 2} = 0.023 \text{ sec.}$$

· ·cording to Hikida (op. cit), 90% of the initial surface temperature rise is
.ssipated within 0.002 seconds, by conduction into the mass of propellant. His
analysis presumed a perfect insulator on the surface immediately after passage of
the blade. If after 0.023 seconds, only a fractional percentage of the ΔT remained,
it can be seen that in 2600 passages per minute, the cumulative effect can be
quite significant. How imperfect the assumed insulator is, and how much less than
shear stress is imposed, both make the situation less serious than assumed. The
fan effect of the cutter may even overcome the heating, but again, it is not
rigorously analysable.

Other evaluations are plausible, i.e., total heat generation, and cumulative effects
as a function of chip, or cut, depth. But they are equally nebulous, due to the
uncertainties outlined above. One can calculate that the total heat generated,
Q, is from 5 to 500 Btu/min. depending on whether there is 0.025 or 2.5 in.^{2} surface
contact by the cutter.

It is my conclusion that dry machining is a process that must ultimately be
qualified empirically. Even then, I recommend against it because of the potential
for foreign objects remaining undetected and for tool failure. It is only at very
low speeds and short cutting durations that one can have confidence that energy
densities and magnitudes are well below the analytically hazardous level.

D. W. Kase

DESCRIPTIONS OF COMPOSITE
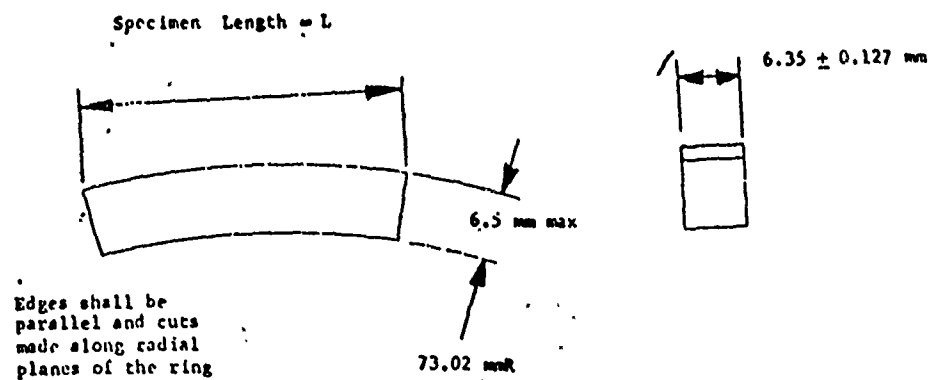
CASE TEST SPECIMEN

AND TESTS

C-140

Specimen Length = L

6.35 ± 0.127 mm

6.5 mm max

Edges shall be
parallel and cuts
made along radial
planes of the ring

73.02 mmR

FIG. 1 Horizontal Shear Test Specimen (Ring Specimen).

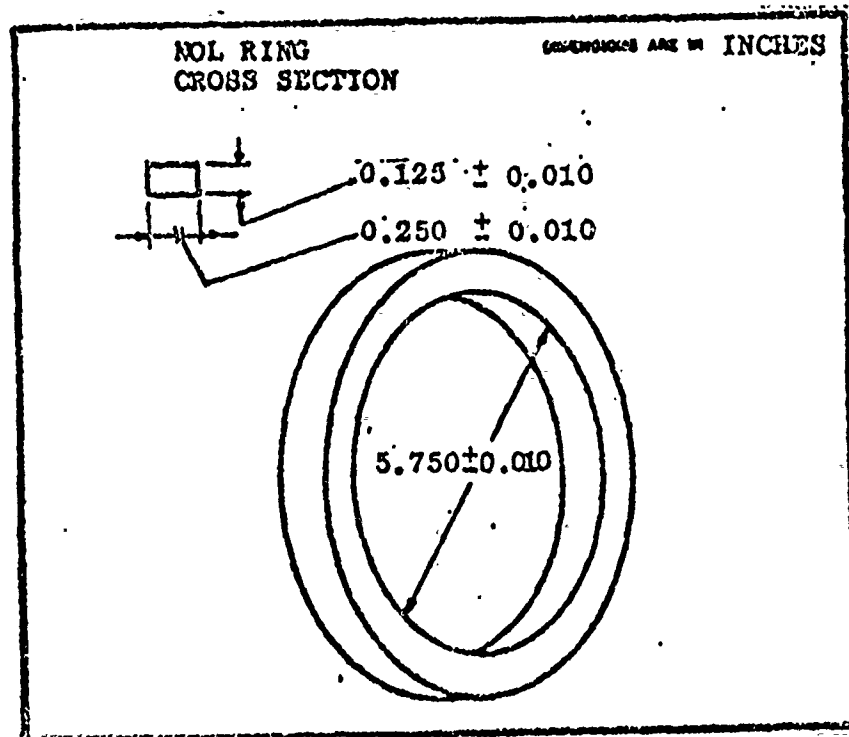NOL RING
CROSS SECTION

DIMENSIONS ARE IN INCHES

0.125 ± 0.010

0.250 ± 0.010

5.750±0.010

Figure 2   NOL Ring

4.5.6   Shear strength. Shear strength shall be determined in accordance with the following.

4.5.6.1   Specimen   fabrication.   Specimens   shall   be fabricated from a NOL ring as follows:

    a.   Assemble cleaned NOL ring mandrel. If the mandrel is not Teflon-coated, apply suitable silicone mold release agent such as MS 122.

    b.   Use NOL ring winding apparatus. Set controls to provide the following:

        (1)   A winding tension of 10 ± 1 pounds at the spacer guide

        (2)   A wrapping rate of 10 to 25 revolutions per minute (rpm)

        (3)   A traverse of the width of the mandrel within 3 to 5 revolutions of the mandrel.

        (4)   Shut down after a total of 46 revolutions have been completed.

    c.   Place sample ball on tensioner, and unspool the roving, passing it through the delivery device onto the mandrel end for ten revolutions in order to hold the roving in place under tension before fabricating test rings.

d. Begin winding the test ring specimens. Wind 46 revolutions, maintaining winding tension from start to end of each specimen fabrication.

e. After test specimens are wound, wind a final tie-off ring (to hold roving under tension during cure) onto the end mandrel using 10 roving revolutions.

f. After winding, maintain the NOL ring at 75 + 10 degrees F for 72 + 10 hours before curing.

g. Mount the mandrel on an oven rotisserie.

h. With the rotisserie turning a minimum of 3 rpm, cure the ring at 210 + 10 degrees F for 8.0 +1, -0 hours.

i. Disassemble the mandrel, exposing the wound ring (with extruded resin) on the central plate. Trim the ring by machining to the required outside diameter (6.005 + 0.005 inches).

4.5.6.2   Test procedure.

a. Using the NOL ring fabricated as specified in 4.5.6.1, radially cut one section out of the ring at points on a chord 1/2 inch.long intersecting the outside diameter. Mount the open ring in a specimen-cutting fixture, and secure ring with clamp. Set fixture to cut specimens 0.635 + 0.010 inch chord length of the outside diameter. Discard first segment.

b. Cut 10 specimens from the ring.

c. Using a suitable testing machine, stress the specimen in accordance with ASTM D 2344 at a crosshead travel rate of 0.05 inch per minute until failure occurs. Repeat the procedure for nine additional specimens, and report the average of ten test results and coefficient of variation.

d. Monitor the strain pattern for each specimen while performing the shear strength test. Shear failure is indicated by the first peak as indicated by the curve. Any increase in load after the first peak is an indication that the specimen is undergoing compression.

4.5.7   Tensile strength (NOL). Tensile strength shall be determined in accordance with the following.

4.5.7.1   Specimen fabrication. Specimens shall be fabricated and cured as described in 4.5.6.1 except 20 revolutions shall be used.

4.5.7.2   Test procedure (for Type I material only).

a. Using a suitable testing machine, mount the NOL ring specimen and test per ASTM D 2290, procedure A, with modifications as indicated herein.

NOTE: Fiber termination points in the ring should be oriented at 3 o'clock prior to testing.)

b. Repeat the procedure for nine additional specimens, and report the average of 10 test results and coefficient of variations.

c. Calculations:

Calculate apparent tensile strength as follows:

$$S = \frac{P}{A}$$

Where: S = fiber tensile strength (psi)

P = maximum load (lb)

A = area of fiber* = 0.0205808 (nominal value)

* Area of the fiber in the ring is based upon 19 revolutions although the ring is wound with 20 revolutions. In order to minimize the chance for the roving to unwind during testing, the part of the ring with only 19 revolutions (between the start/stop tabs) shall be oriented at 3 o'clock.

4.5.8 Mechanical strength (mean hoop fiber strength at burst (Type I material only)).

NOTE: This test will be conducted by Thiokol on one bottle set per lot.

a.   Fabricate three 5.75-inch-diameter bottles per ASTM D 2585 procedures with modifications as specified herein.

b.   The design of the 5.75-inch bottle shall be as specified in table VI.

TABLE VI. 5.75-INCH BOTTLE DESIGN

Diameter	5.75 inches
Process	Roving impregnated with resin system (see table I)
Burst mode	Hoop
Polar & hoop plies	2 polar, 3 hoop
Ends/inch	37.865 polar, 40.18 hoop
Rovings/band	1 polar & hoop
Stress ratio	0.851
Wafers	None.
Resin percent	30 ± 2 polar & hoop
Winding tension	10 pounds polar & hoop
Wind angle (deg)	12.0
Cure cycle	8 +1, -0 hours at 210 ± 10 degrees F

c. Test per ASTM D 2585 at 70 ± 10 degrees F, and record maximum pressure achieved (P).

d. Calculation:

Calculate the fiber strength as follows:

$$S = (174.755) P$$

Where: S =   fiber strength in psi

P =   burst pressure in psi

The mean fiber strength from these three
samples shall be greater than the value
specified in table III. If the coefficient of
variation is greater than 3.0 percent, the
test shall be rerun.

4.5.9   Glass transition temperature. The glass transition
temperature shall be determined as follows:

4.5.9.1   Specimen preparation.

a. Using a Teflon-coated Reometrics Dynamic
Spectrometer (RDS) with grooves 0.498 + 0.002
inch wide and approximately 7 inches long,
wind 11 plies of roving with a winding tension
of 10 + 2 pounds.

b. Wrap the mandrel with one layer of green
release cloth, and vacuum bag the mandrel
throughout the cure.

c. Cure at 210 + 10 degrees F for 8 +1.0 -0.0
hours.

d. Remove the sample from the mandrel, and sand
off resin flashing, if any is present.

e. Using an abrasive cut-off wheel, machine off
approximately 1 inch from the end, and cut the
specimen to 2.5 + 0.1 inches long.

4.5.9.2 Test procedure.

a. Set up a calibrated Rheometrics Dynamic Spectrometer with a 10,000-gram transducer as follows:

(1) Mode: Temperature sweep

(2) Test geometry: Rectangular torsion

(3) Beginning temperature: 40 degrees C

(4) Last temperature: 180 degrees C

(5) Degrees per step: 5 degrees C

(6) Thermal soak time: 1 minute

(7) Correlative delay: 3 seconds

(8) Strain: 0.5 percent

(9) Frequency: 6.28 radians per second

(10) Plot: G", G*, Tan delta vs temperature

(11) X axis zero: 40

(12) X axis maximum: 180

(13) Y axis zero: 10 to the 6th power (5-cycle graph paper) or 10 to the 7th power (4-cycle graph paper)

(14) Y axis maximum: 10 to the 11th power

(15) Print: G', G", G*, Tan delta, torque, temperature

(16) Page title: Sample name, date, laboratory test identification number.

b. Place a normal load tension of 20 percent upon the sample upon initial loading. After starting the test, do not adjust the normal load.

4.5.9.3 <u>Test interpretation and reporting</u>. The glass transition temperature shall be interpreted and reported as follows:

a. Interpret the glass transition temperature (Tg) from the tan delta and G" versus temperature plots as the intersection of the tangents to the curve from both sides of the maximum in the curve.

b. Using a straightedge, draw the tangent through the maximum number of points as closely proximate to, but probably not including, the maxima points in the curve.

c. If there is no maximum in the curve, draw the lines through the points on both sides of the first abrupt change in the slope of the curve.

d. Report Tg tan delta and Tg G" to the nearest 0.5 degree C, and submit a copy of the RDS data to Thiokol with the test results.

APPENDIX D

PHASE IV INTERIM REPORT

PROGRAM PLAN

FOR

FULL SCALE DEMONSTRATION OF COMPOSITE
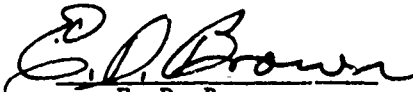
CASE SALVAGE TECHNIQUES

PROGRAM PLAN

FOR

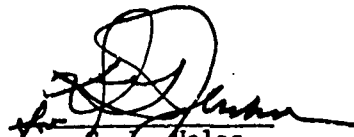FULL SCALE DEMONSTRATION OF COMPOSITE
CASE SALVAGE TECHNIQUES

SUBMITTED TO

DEPARTMENT OF THE AIR FORCE
HEADQUARTERS, AIR FORCE FLIGHT TEST CENTER
DIRECTORATE OF CONTRACTING (PKRA)
EDWARDS AFB, CA 93523

In Accordance With
Contract F04611-81-C-0001
CDRL No. 6

22 February 1982

_____   _____
E. D. Brown   G. L. Hales
Principal Investigator   Program Manager

THIOKOL/WASATCH DIVISION
A DIVISION OF THIOKOL CORPORATION
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Publications No. 82104

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ILLUSTRATIONS

TABLES

1.0 INTRODUCTION

The increasing cost of filament wound structures combined with the long lead time required for new cases has prompted the initiation of a Composite Case Salvage Study Program.

The program was initiated by AFRPL under Contract F04611-81-C-0001 on 15 November 1980. The program was divided into four phases. The Phase I effort consisted of an assessment of existing technology. Phase II effort consisted of the development of a cost model and the comparison of the feasibility and cost effectiveness of various salvage methods. Phase III, Laboratory Studies, consisted of testing propellant removal techniques of both Class 1.1 and Class 1.3 propellants identified in the two previous phases. Phase III also consisted of determining the impact that the removal techniques had on both insulation systems and case materials.

The results of the Phase I, II, and III studies show that the safest, most cost effective way to reclaim composite cases loaded with Class 1.3 propellant is to employ hydromining. Wet machining removal was identified as the safest method of removing Class 1.1 propellant.

During the oral presentation of the Phase III results, AFRPL directed Thiokol to address reclamation of MX Stages I, II, III in addition to Minuteman III Third Stage motors in the Program Plan, Phase IV.

2.0 OBJECTIVE

The objective of this program is to demonstrate that Class 1.3 propellant can be successfully removed for a Minuteman III Third Stage composite motor case without compromising the suitability of the case for reloading and reuse.

The demonstration will be accomplished using 3 Minuteman III Third Stage motors furnished as GFM by AFRPL.

3.0 SCOPE

The program is designed to provide the Reclamation of full scale Composite Cases by removing propellant from the Minuteman III, Third Stage, the MX Stages I and II, and the MX Stage III rocket motors and demonstrating that they are functional for further use. The program provides information for removal of Class 1.3 propellant and Class 1.1 propellant from Solid Propellant Motors utilizing Composite Motor cases. The program provides post propellant removal processes to provide a clean, dry, and sound case for further testing. The cross-combination program provides for the salvage of four distinctly different but related composite rocket motor cases (Table I).

3.1 RECLAMATION OF CASES WITH CLASS 1.3 PROPELLANTS

3.1.1 Minuteman III Third Stage

Reclaiming Minuteman III Third Stage fiberglass composite rocket motor cases loaded with Class 1.3 propellant will be evaluated with propellant removal, case clean up and drying, propellant waste disposal, and case testing to verify that the reclaimed case is sound and functional for further processing (see Figure 1). Qualification in a Weapons System is not included. Three motors will be processed. One motor will be hydroburst, one will be structurally loaded to failure, and the last will be loaded with propellant and static tested.

3.1.2 MX Stage I and II

The MX Stage I and II Kevlar composite rocket motor cases also contain the Class 1.3 type propellant, and the program will evaluate propellant removal, insulation cleanup, case drying, and propellant waste disposal methods. The MX Program at the present time is still in the development stage and the cases are relatively new. Extensive testing to prove the structure of the reclaimed case would not be required providing the Minuteman III Third Stage case meets all of the above testing requirements.

3.2 RECLAMATION OF CASES WITH CLASS 1.1 PROPELLANT

Composite cases that contain explosives Class 1.1 propellant are considered to be borderline from the standpoint of cost effective propellant removal and case reclamation methods. Coupling the low cost effective position

TABLE I

SUMMARY OF VERIFICATION PROGRAM PLAN FOR SALVAGE
OF SELECTED COMPOSITE CASES FROM SOLID PROPELLANT ROCKET MOTORS

PROGRAM	SCOPE	SAFETY	PROPELLANT REMOVAL	CASE PREPARATION PRIOR TO REUSE	PROPELLANT DISPOSAL	CASE DISPOSITION
Minuteman III Exp. Class 1.3	Three 3rd Stage Rocket Motors: One to be cleaned out and hydroburst. One to be cleaned out and structural load tested. One to be cleaned out, reloaded with AP/Al solid propellant and fired.	The propellant removal system is proven and 2 3rd Stage Minuteman units have previously been hydrotested and insulation reviewed. Low risk safety operation.	Bulk propellant removal with hydromining 150°F water. The remaining propellant removed with hydromining 180°F water at 45° angle to insulation.	Remaining liners are buffed out if case insulation is buffed, cracked up, patches as necessary, flaps installed, cases cleaned and ready for propellant casting.	1. The burning pit area. ALTERNATE: The AP saturated liquor is sent to a crystallization AP Recovery Plant. Sludges are sent to a hazardous land fill.	One case will be hydrotested to 1.1 MEOP then hydroburst. One case will be hydrotested and structure in the compression mode per acceleration during flight. One case will be sized and loaded with AP/Al and fired after which the case will be evaluated for insulation loss and accumulated structural change.
ME Exp. Class 1.3	One motor for reuse and feasibility study.	The HTPB-AP propellant under development for ME have safety parameters that are similar to Stage III propellant. Low risk safety operation.	Bulk propellant removal with hydromining 150°F water. The remaining propellant removed with hydromining 180°F water at 45° angle to insulation.	Same as above.	Same as above.	The in process loss salvaged case would be used for additional development motor work or is the early part of production, would be used as a quality round or other static firing test.
Minuteman II Exp. Class 1.1	One motor for reuse and feasibility study.	The Nitroglycerin double base propellant presents unique safety problems in handling and removal operation based on the disposal of a very high propellant utilization based on Shelfield, England's incident.	Least hazardous method – the mechanical cutting block and outing the last 1/4" of material from the insulation.	To remove the epoxy asbestos liner system by hand buffing underwater.	The explosives to be treated with sodium hydroxide to neutralize the nitroglycerin and the system taken to open air burning pit areas.	Same as Minuteman III, Explosive Cl. 1.3 above.
ME Exp. Class 1.1	Stages I and II – in process loss units loaded with the respective propellant. Motor cases used for development or related testing rounds.	ME Stage III in process loss propellant may be loaded with cross-linked double base NG. NG type insulation. The system is less sensitive to ignition and less likely to ignition double base propellant. Testing is composite case reclamation procedures indicate medium risk.	Bulk propellant removal with hydromining 150°F water. The remaining propellant removed with hydromining 180°F water at 45° angle to insulation. ALTERNATE: Same as for Minuteman, Exp. Class 1.1 above.	Same as Minuteman III, Exp. Cl. 1.3 above. ALTERNATE: Same as Minuteman II, Class 1.1 above.	Same as Minuteman II, Cl. 1.1 above.	Same as ME, Exp. Class 1.3 above.

o   RECEIVE LOADED CASE

o   VISUAL INSPECT

o   X-RAY INSULATOR CASE BOND

o   INSTALL CASE PROTECTIVE COVERING
    AND TOOLING

o   HYDROMINE - REMOVE PROPELLANT AND LINER

o   DRY CASE

o   INSPECT CASE VISUAL/X-RAY BOND FOR
    INSULATOR

o   HYDROTEST

o   DRY CASE (NOT REQUIRED FOR CASE NO. 1)

CASE NO. 2   CASE NO. 1   CASE NO. 3

INSTRUMENT AND SET UP   INSTRUMENT AND SET UP
FOR STRUCTURAL TEST   FOR HYDROTEST AND
                              HYDROBURST
STRUCTURAL LOAD TEST
                              HYDROBURST (TO BE DONE
EVALUATE DATA AND   IN CONJUNCTION WITH
REPORT   CASE ACCEPTANCE
                              HYDROTEST ABOVE)

                              EVALUATE DATA AND   VISUALLY INSPECT CASE
                              REPORT
                                                        INSTALL FLAPS

                                                        LINE CASE
                                                        CAST AND CURE MOTOR

                                                        X-RAY LOADED CASE

                                                        WEIGH MOTOR

                                                        ASSEMBLE MOTOR FOR
                                                        STATIC TEST

                                                        FINAL INSPECTION
                                                        AND BUY-OFF

                                                        TRANSPORT TO AIR FORCE
                                                        TEST FACILITY

                                                        STATIC TEST

                                                        RETURN TO TC FOR
                                                        POSTFIRE ANALYSIS

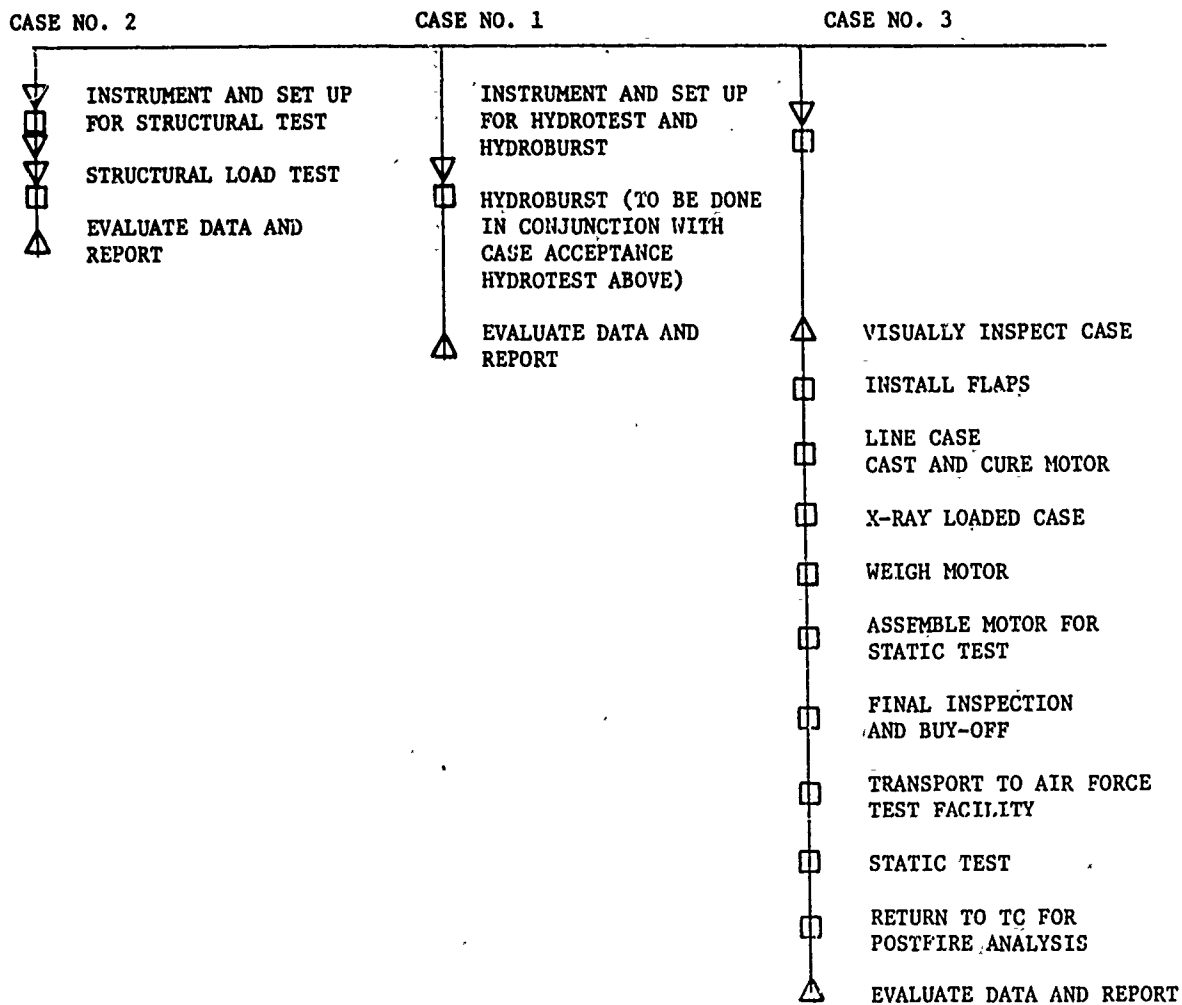   EVALUATE DATA AND REPORT

Figure 1.   Minuteman Stage III Case Reclamation Flow Sheet

with high hazard material handling provides a reclamation concept of low value. However, costs are not always the driving parameter when a program schedule has to be met. Therefore, the MX Stage III containing Class 1.1 propellant will be evaluated.

3.2.1 MX Stage III

The MX Stage III motor is loaded with a crosslinked, doubled based propellant that contains both HMX and NG. Since an inprocess loss is always considered in a program, it could become necessary to reclaim an MX Stage III case to meet schedule requirements. The program will provide the propellant removal, propellant waste handling, case cleaning and drying process, and the testing sequence for further use in a program.

3.3 PROGRAM MASTER SCHEDULE

The program schedule (Figure 2) depicts an orderly outline of the effort required to demonstrate the reclamation and testing of three Minuteman III Third Stage motor cases. The program is divided into three phases:

      Phase I   - Motor Washout and Acceptance Testing

      Phase II - Reclaimed Case Verification Testing

      Phase III - Motor Fabrication and Test

Total program length is 20 months, with 16 months of reclamation, fabrication, testing, and evaluation followed by four months to finalize the final report. During the Phase I motor washout and acceptance testing, the reclaimed case configuration will be established by Engineering. Reclamation process standards will also be prepared by Engineering. This will establish the critical process limits such as water pressures, temperature, dwell times, etc., for the hydromining operation. Tooling required to protect the case during hydromining will be designed and fabricated.

The manufacturing and inspection planning will be formalized for the following:

      1.   Motor Receipt

      2.   In-Process Handling

      3.   Pre-Test Inspect

      4.   Hydromining Processing

      5.   Post Washout Operations and Inspection
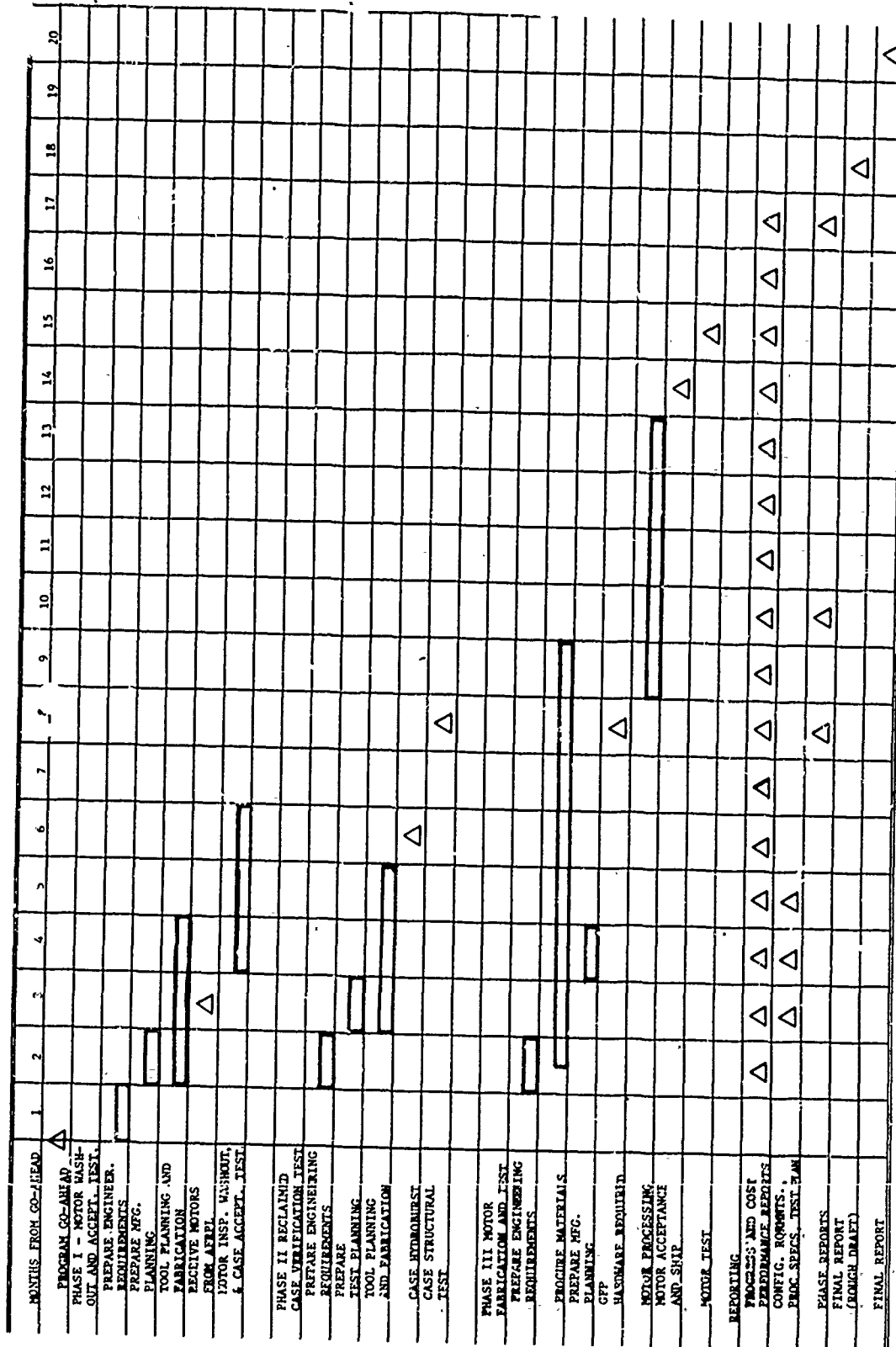
      6.   Hydrotest

Figure 2. Minuteman III Third Stage Motor Case Reclamation and Test Demonstration Schedule

The three Third Stage motors will be required by the third month of the program. Pre-test inspection, including X-ray of the insulator to case bond, will be accomplished prior to hydromining. Preparation for propellant hydromining, hydromining, post-hydromining cleanup, and drying will be accomplished in accordance with the engineering requirements and manufacturing processes previously established. This will be followed by case inspection including X-ray and nondestructive hydrotesting in accordance with established Third Stage procedures. This Phase I effort is estimated to cover six months to reclaim the three cases. Structural verification testing is planned for two of the cases from Phase I under the Phase II portion of the program. A test configuration will be established, and two test plans and instrumentation drawings will be prepared by Engineering for the case to be hydroburst and the second to be subjected to structural testing. Acceptable limits will be established from data on file from new case testing accomplished during Minuteman III Third Stage motor development and production testing. Tooling planning will identify available tooling stored by the Air Force from the Minuteman III Third Stage program for cleanup and use under this phase. Additional tooling required will be designed and fabricated.

Detail test planning will be prepared for instrumentation, test setup, test, and post-test evaluation based upon the engineering requirements established. Hydroburst of the first motor is planned for the sixth month followed by structural testing of the second motor in the eighth month as shown.

Phase III effort consists of loading the third reclaimed case, assembly of the motor to the configuration to be established, and shipping the accepted motor to the Air Force for static test. Engineering will be released to define the test motor configuration including bills of material, drawings, specifications, and test plans including post-test requirements. A procurement plan will be released to authorize purchase of flap material, propellant, and liner materials, etc., for standardization and motor requirements as established by Engineering. Lead time on the propellant materials is estimated at seven and a half months as shown.

It is assumed that the subsystems required for motor testing such as the S&A igniter assembly, nozzle assembly, and AOTTS, LITVC roll control assemblies, if required, will be furnished GFP from Air Force inventory. Costs of

these items would be prohibitive for subcontractor start-up and fabrication for one unit.

Manufacturing and quality planning used in the Third Stage production program will be used in preparing the planning for this motor in accordance with the engineering requirements. It is assumed that Third Stage production tooling currently being stored by the Air Force will be available for use on this program.

The GFP items identified for motor assembly will be required by the seventh month of the program. Motor fabrication is estimated to cover five months with review and acceptance by the Air Force prior to shipment during the 14th month and test in the 15th month. The motor will be returned to Thiokol after test for post-test evaluation and analysis.

Monthly progress and cost performance reports will be submitted as shown through the program duration. Three sets of design configuration and test plan submittals are planned as shown. The Phase I requirements will be submitted in the third month followed by the Phase II in the fourth month and Phase III in the fifth month as shown. A review of each phase will be presented at the completion of each phase in the 8th, 10th and 17th months, respectively. A rough draft of the final report will be submitted for AFRPL comments and approval prior to release of the final report in the 20th month.

A separate schedule for reclamation of a case from an MX motor is not included. A review of the necessary requirements planned for reclaiming an MX First or Second Stage motor would indicate that the time involved for one motor of either stage would fall in line with the six month time span shown for the reclamation of three Minuteman motors as shown in Phase I.

A requirement for separate facilities, tooling, and processing approach for an MX Third Stage motor containing Class 1.1 propellant precludes realistic scheduling without considerable more planning than is available as a result of the current program.

4.0 WORK TO BE ACCOMPLISHED

This overall program task provides in detail the methods of propellant removal, insulation clean up and case drying, waste disposal, and testing required to reclaim a composite case for program use. The criteria for composite case salvage is shown in Table II.

4.1 RECLAIM CASES LOADED WITH CLASS 1.3 PROPELLANT

Rocket motor composite cases loaded with Class 1.3 propellant include the Minuteman III Third Stage and MX Stage I and II systems. Each represents a Weapons System that is deployed or in development. The studies conducted in the case composite procedure development program have shown that methods for reclaiming each are feasible and cost effective.

4.1.1 Comparison of Minuteman III Third Stage and MX Stages I and II

For all situations, the Minuteman III Third Stage and MX Stage I and II are very similar inasmuch as the propellants contain approximately 70% ammonium perchlorate. The binder system in the Minuteman Third Stage motor is a carboxyl terminated polybutadiene (CTPB) while the MX Stage I and II binders are hydroxyl terminated polybutadiene systems (HTPB). Both propellants are effectively hydromined, as shown in the Case Salvage Procedures Development Program. Cutting rates on both Minuteman and MX are comparatively high. The Minuteman composite case is fiberglass whereas the MX cases are Kevlar fibers. Both cases contain materials that are compatible with the hydromining process as long as the internal insulator is left intact, and both need to be cleaned and dried to recover their initial physical properties. The larger size and high cost of the MX cases make them very cost effective for salvage with the salvage operation costing about one tenth the original cost of the composite case whereas with Minuteman Third Stage the fabrication is much closer to the cost of salvage operation.

4.1.2 Method of Propellant Removal

The hydromining facility at the Wasatch Division will be used to remove propellant from either MX Stage I and II or Minuteman III Third Stage rocket motors. The motors will be mounted in position on a track in the hydromining facility and the nozzle holding tools positioned for each size of motor (see Figure 3). The operation will be conducted by using 3,000 psi water at 150°F.

TABLE II

SELECTION CRITERIA FOR SALVAGING

| Rank	Parameter	Limiting Factors – Drivers
1	Safety, Personnel, Facilities	No risk to personnel injury is allowed. Risk to a 10^{-5} probability level (hazardous analysis)
2	Cost	To the limit of fabrication Fabrication Cost > Salvage Costs (May include facility cost for fabrication where required.) (To include additional facilities and qualifications where required.)
3	Effect on Case and Insulation Structural Integrity	Case structural integrity will not fall below original design required margin of safety.
4	Effect on Case Reloadability	The insulated case will be capable of being processed through propellant loading methods.
5	Disposal of Waste Products	Cost, personnel hazards, and acceptable requirements must be maintained.
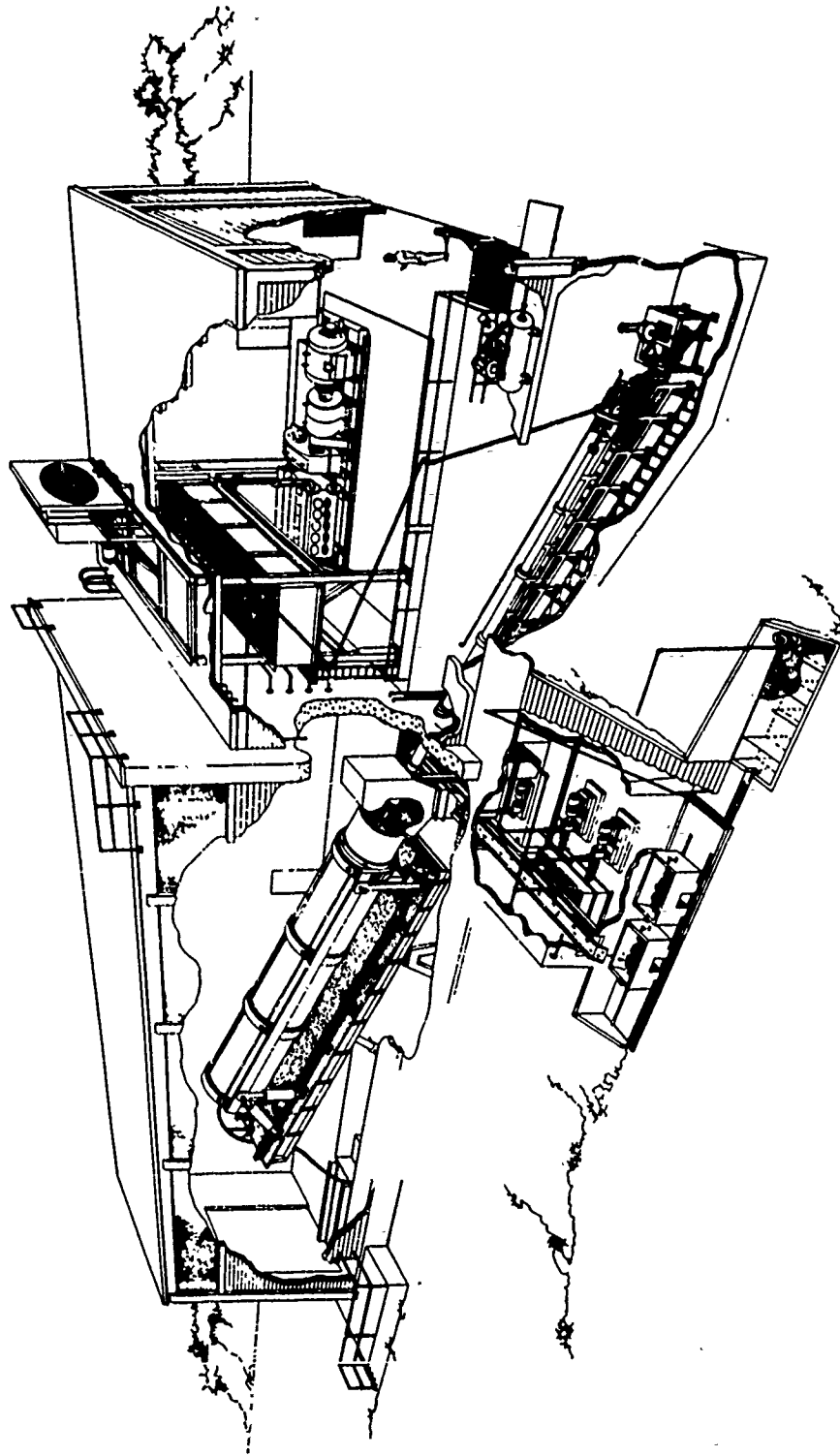
Figure 3. Wasatch Division Case Reclamation Facility

Several passes through the motors will be made to remove the bulk of propellant and will leave approximately 1 in. of propellant on the insulator, wherein the pressure will be adjusted downward to approximately 2,000 psi and the water temperature adjusted to 180°F. The angle of cut will be changed to 45 deg with respect to the insulator surface. The insulator will be washed clean with this system based on data from Phase III of Composite Case Reclamation Procedure study. After the propellant has been removed, the insulator surface will be examined for residue and the case and rubber system dried for further processing. During propellant removal, fiberglass or Kevlar composites and metal parts will be protected from water by providing special tooling and using waterproof plastic films to cover the external surfaces of the motor.

4.1.3 Waste Propellant Disposal

During hydromining of propellant, the sludge from the propellant will be analyzed for ammonium perchlorate (AP) content; and, when it is below 5% AP, it will be removed to the sludge disposal areas. The water system is maintained at 150°F to pride a high AP dissolution as well as to enhance the cutting operation. The AP solution will then be recycled to the crystallization areas in the newly fabricated 100 lb per hour propellant-AP recycling facility where the AP will be crystallized and packaged for the AP salvage market. An alternate to the above mentioned reclaiming the AP is to put the solution in a solar pond.

4.1.4 Case Preparation

The composite cases, be they Kevlar for MX or fiberglass for Minuteman, will be further examined for complete propellant and liner removal. In those areas where liner (propellant bonding media) is still present, the rubber insulator will be buffed to provide a clean surface for future bonding and lining applications. The stress related flap remnants will be removed and the flap bonding area buffed in preparation for new flap insulation. The cases at this point will be readied for the dry out process and dried for 48 hours at 135°F, after which the case will be hydrotested and ready for hydroburst, structural load testing, or propellant loading and static testing.

4.1.5 Testing Requirements to Demonstrate the Functional Use of Reclaimed Composite Cases for Program Use

4.1.5.1 Minuteman III Third Stage

Three Third Stage Minuteman cases will be salvaged by hydromining the propellant as described previously for use in the demonstration test phase of the program. Two case structural tests will be performed to demonstrate structural integrity of the salvaged cases; one case will be hydroburst; one will be subjected to a flight load test; and one will be loaded with propellant and static fired.

4.1.5.1.1 Hydroburst Test

One Minuteman Third Stage case will undergo a hydroburst test in accordance with Test Plan TPIII-020 after the normal hydroproof test required for production cases. The test arrangement is shown in Figure 4.

4.1.5.1.2 Structural Test

One salvaged Minuteman case will be structurally tested in accordance with TWR-4489, Test Plan for Flight Loads of Third Stage Minuteman Case. The structural test arrangement is shown in Figure 5.

4.1.5.1.3 Motor Static Test

The third case will be loaded with ANB-3066 propellant after successfully passing the hydroproof test. The motor will be assembled to the 1147372-91 Rocket Motor Final Configuration and tested in accordance with TWR-4269, General Test Plan Third Stage Minuteman III Production Quality Assurance (PQA), at Arnold Engineering Development Center (AEDC), Tullahoma, Tennessee.

4.1.5.2 MX Stage I and II

In the event a MX composite case requires salvaging, the hydromined case would be carefully inspected for case fiber damage and internal insulation damage. Any internal insulation damage would be repaired using standard repair procedures. A second hydroproof would not be performed in the event of case fiber damage unless dictated by Material review action.

4.1.6 Facilities

Thiokol/Wasatch has a complete facility capable of the reclamation of composite cases. Some Minuteman III Third Stage motors have been processed

OUTLET PORT ← → INLET PORT

HYDROTEST
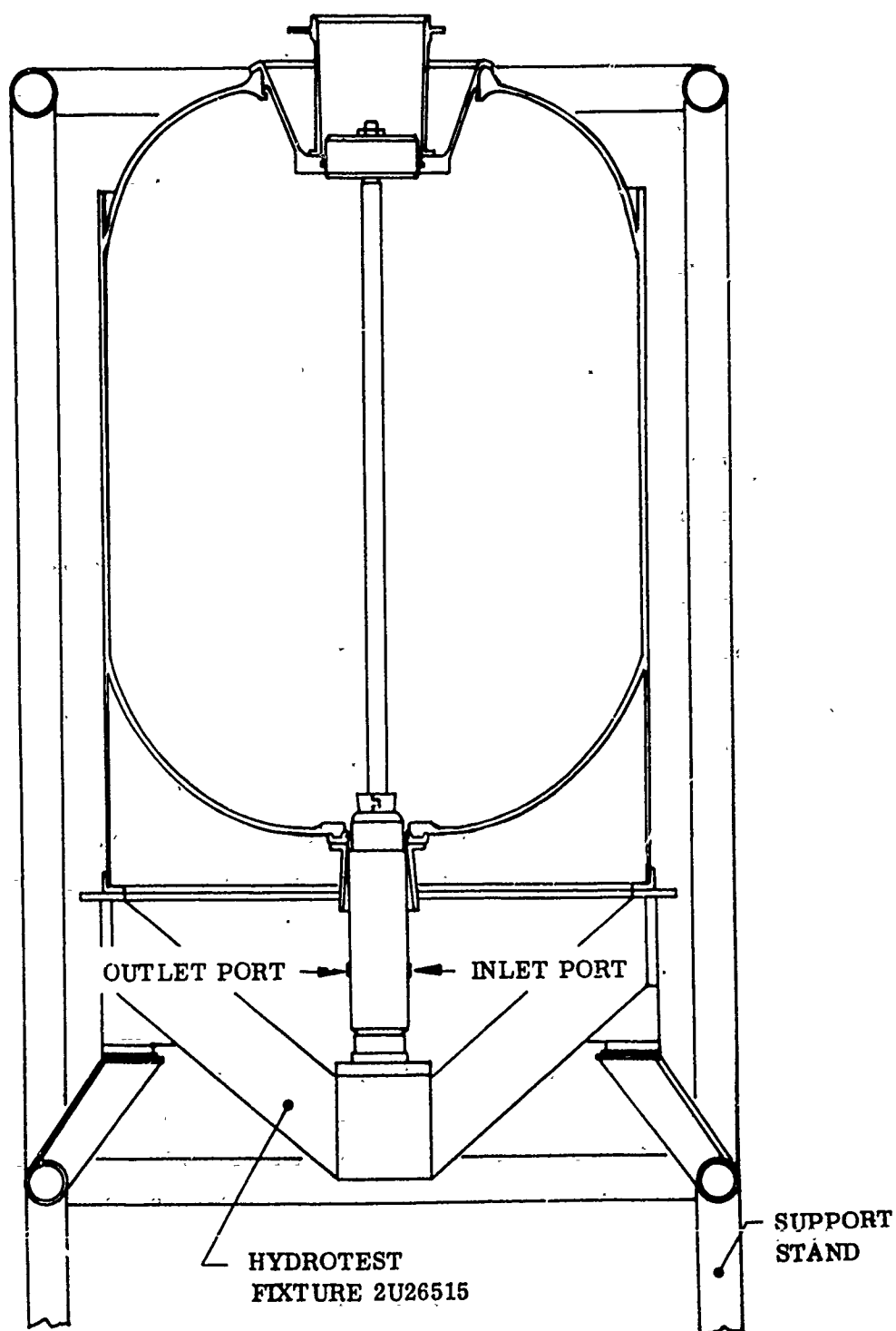FIXTURE 2U26515

SUPPORT
STAND

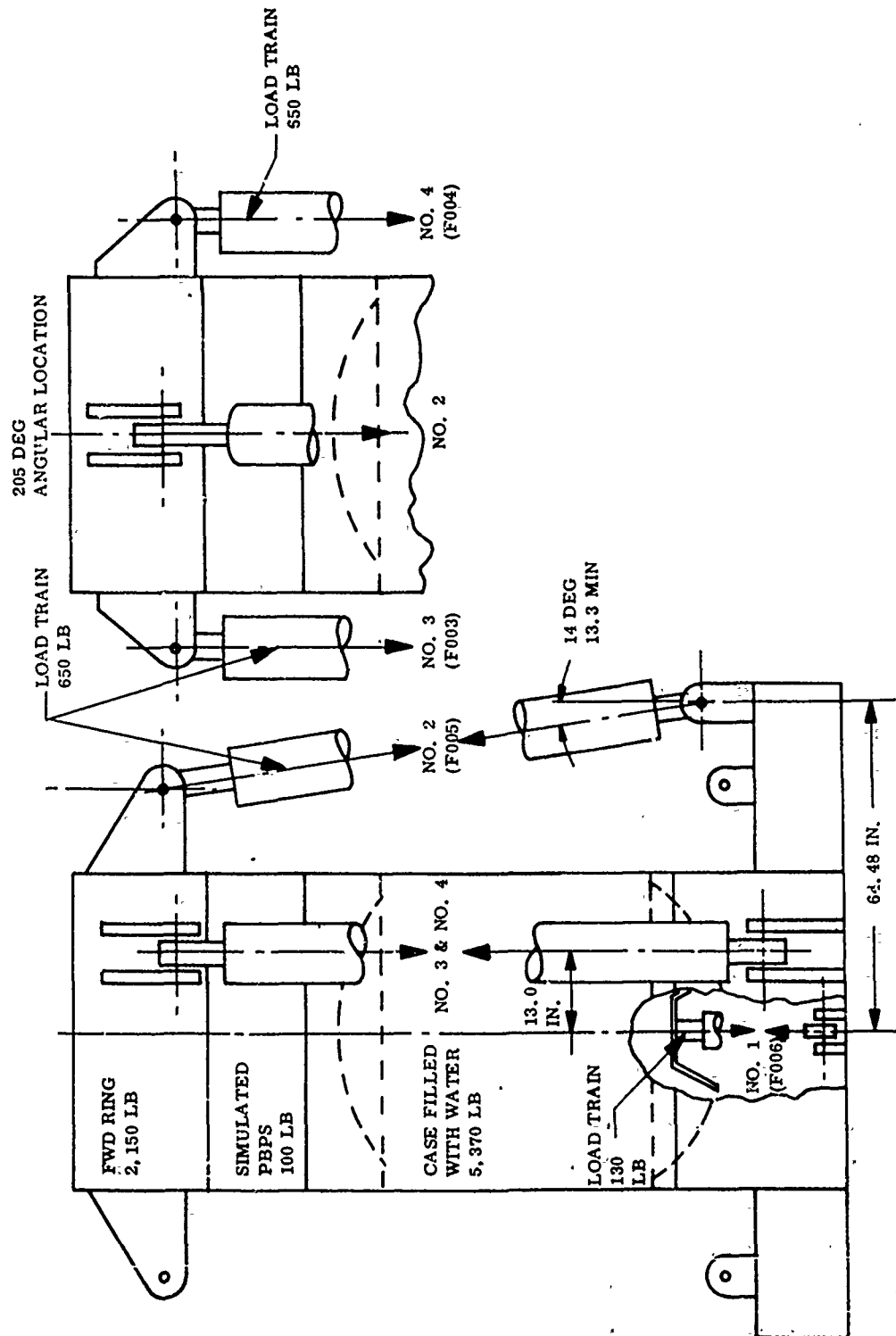Figure 4.   Sketch of Test Setup (Full Scale)

Figure 5. Flight Load Phase II Test Setup

through the hydromining facility. Work done by Thiokol to analyze the feasibility and costs for a planned Minuteman III Third Stage Retrofit Program has verified these facilities. Only minor tooling is required to adapt the MX Stages I and II to the reclamation process. Building M-115 (Figure 4) is currently being used to clean up insulator systems on retrieved Space Shuttle SRMs. A Kevlar case was reclaimed from a C-4 Trident rocket motor. This motor was successfully processed through the facility and subsequently loaded with inert propellant. This past history demonstrates a capability of handling and protecting Kevlar composite systems. Thiokol/Wasatch has proven methods for the disposal of hazardous waste at present and meets all EPA requirements as a licensed hazardous waste handler and disposal site. We are currently constructing a 100 lb per hour propellant reclamation facility to salvage AP from Class 1.3 propellants.

4.2 RECLAIMING COMPOSITE CASES LOADED WITH CLASS 1.1 PROPELLANT

4.2.1 Review Cost Trade Off Analysis

The cost analysis conducted in Phase II of the Composite Case Salvage Procedures Program indicates that Minuteman II Third Stage reclamation costs exceed the cost of fabricating a new case. It is proposed to finalize cost analysis on Minuteman II Third Stage and demonstrate total cost trade off packaging to verify the past cost analysis and trade offs. No further work would be done. The program cost analysis does indicate that the higher cost Kevlar cases could possibly be cost effective to reclaim the composite cases. A cost trade off analysis will be finalized for MX Stage III from the viewpoint of case salvage vs case fabrication.

4.2.2 Salvage Hazards Review

The Safety Hazards data is complete in the situation where one considers case salvage of Minuteman II Third Stage systems. At Sheffield, England, an incident occurred while hydromining double based propellant, and the hazards analyses verify that the double based propellant is a high risk. On the MX Stage III motor system a hazards analysis will be completed and a trade off made to use hydromining or machining to remove propellant from the Kevlar case.

4.2.3 Propellant Removal Methods

The propellant removal methods can be hydromining or machining. The major difference between hydromining of Class 1.1 and Class 1.3 propellant is the type of facility to be used. Since there is a higher risk in Class 1.1 propellant removal by hydromining, a small, temporary and expendable facility would be required for this approach. With this being the only major difference, one can use the logic from previous sections for hydromining Class 1.3 propellant. The propellant removal method most attractive to remove the Class 1.1 propellant from the MX Stage III case based on these studies is machining. At completion of the safety and cost analysis, machine cutting tools similar to those defined in Phase III of the Composite Case Salvage Process Program would be fabricated and set up in a remote and expendable facility wherein the propellant would be cut from the case. After the bulk of the propellant is removed to less than 1 in. thickness, hydromining techniques would be required to remove the remainder of the propellant from the insulation.

4.2.4 Waste Propellant Disposal

The waste propellant would be collected in large containers and moved to the disposal area. The nitroglycerin contaminated water would be treated with hydroxyl to render it safe to handle. The waste disposal ponds for collecting the propellant waste water and propellant sludge would be dried using solar evaporation, after which they would be burned to render them safe.

4.2.5 Insulation Cleanup

The MX Stage III case, after propellant removal, would be moved to an area where the remaining powder embedment lining system, the epoxy binder, could be removed from the insulator. Where powder embedment is still bonded to the system, water buffing can be used to clean the rubber, after which the case would be dried and the remaining rubber buffed and cleaned. The stress release flap remnant would be removed and bonding areas buffed. At this point the case would be inspected to the original design drawings. After the insulation is cleaned and processed to the stress release flap installation, the case would be dried for 48 hours at 135°F.

4.2.6 Facilities for Class 1.1 Propellant Removal

As described earlier in the propellant removal methods, the facilities required for removal of Class 1.1 propellant for a composite case would include a temporary structure where the structure, the tooling, and the hold-down stands are all remotely operational and expendable. Special designs and constructions would be made. The propellant removal facility would be something like a specially prepared Dempsey Dumpster where, as it is filled it would be removed to the solar ponds for evaporation and open air burning. The high pressure water pumps for cleanup of the insulator would be located remote to the actual operation. Control bunkers would be set up to protect personnel.

At the present time there are no facilities in the industry to accomplish Class 1.1 propellant removal techniques.